Client report

ALBERTA RESEARCH COUNCIL

Oil Sands and Hydrocarbon Recovery

ALTERATIONS TO BITUMEN PRODUCED BY THE IN SITU COMBUSTION PROCESS AT THE OXYGEN WOLF LAKE PROJECT, ALBERTA

Final Report

by

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EXECUTIVE SUMMARY

PART I:

Produced oil samples collected over a one year period from well EX-32 at the in situ combustion pilot at Wolf Lake, Alberta were physically and chemically characterized in detail. The properties of these samples were compared to those for oil from a core sample obtained prior to the initiation of the fireflood. The objective of this work was to assess the effects of the in situ recovery process on the quality of the produced oil, with particular emphasis on the subsequent upgrading process.

Upgrading of the produced oil from the in situ combustion field pilot was reflected in the reduced viscosity and density of the whole oils and increases in the amount of material boiling in the naphtha and middle distillate ranges. A concomitant decrease in the wt% of the residue cuts was observed. The absence of an increase in heteroatom or asphaltene content over the portion of the production cycle studied was an auspicious sign for the subsequent upgrading of the produced oil.

The naphtha cut exhibited significant increases in aromatic content early in the production cycle. In addition, there was a sustained shift to lower carbon numbers in the various compound classes. This presence of "new" compounds in the naphtha cut as a result of the combustion process was highlighted by the appearance of C_6 to C_{12} n-paraffins.

The aromatic content of the middle distillate cut also increased early in the production cycle although there was little indication of permanent shifts in compound classes or carbon number distributions. It was the relative growth in the wt% of the middle distillate cut which most affected the quality of the produced oil.

The wt% of the gas oil cuts underwent little change as a result of the fireflood and there appeared to be little relative change with respect to compound classes in the chromatographic fractions. There were, however, shifts to lower carbon number for some of these classes.

The wt% of the residue cuts for the fireflood-produced oils were lower than that of the corresponding cut from the core sample. There was no significant change in the nitrogen or sulfur content of the residue over the time period of the study. In addition, there were negligible increases in the asphaltene and polar components of the residue cuts from the produced oils relative to the core sample.

It is generally concluded that the quality of the oil produced from EX-32 during the one year period of this in situ oxygen combustion process should cause no major problems in the subsequent upgrading of the oil. On the contrary, the properties of the oil suggest that the recovery process itself resulted in a small but significant amount of upgrading.

PART II:

Produced oil samples collected over an eight month period from well EX-6 at the in situ combustion pilot at Wolf Lake, Alberta were physically and chemically characterized in detail. The properties of these samples were compared to both those of oil from a core sample obtained prior to the initiation of the fireflood and those of the produced oil from well EX-32. The objective of this work was to assess the effects of the in situ recovery process on the quality of the produced oil, with particular emphasis on the subsequent upgrading process.

A number of analytical techniques were applied to the whole oils with four samples chosen for a fractionation by distillation and column chromatography. These fractions were characterized in detail by gas chromatography - mass spectrometry (GC-MS).

Relative to the core sample, the whole EX-6 oils produced during the in situ combustion process exhibited a decrease in viscosity which was correlated to an increase in the amount of material in the naphtha boiling range. Unlike the EX-32 produced oils, the EX-6 oils did not show an increase in material boiling in the middle distillate range.

The elemental analyses and acid numbers for the EX-6 oils indicated that the chemical effects due to any low temperature oxidation which occurred as a result of the in situ combustion process were minimal. Based on the analytical results, there was no chemical basis (e.g. an increase in hydrocarbon-based surfactants) to rationalize the "tight" emulsions which these fireflood-produced oils formed.

The GC-MS analyses of the naphtha distillation cuts and the saturate fraction from the middle distillate cuts of the EX-6 oils indicated few significant changes in either the compound class or carbon number distributions. In contrast to the results for the EX-32 oils, there was no chemical evidence to suggest any dilution of the EX-6 produced oils by light material either distilled from near the firefront or produced by thermal cracking at the firefront. Overall, the in situ combustion process caused only minimal chemical changes to the EX-6 produced oils.

In general, the EX-6 produced oils were upgraded relative to the core sample but to a lesser extent than the produced oils from EX-32. The quality of the EX-6 oil produced during the period of the in situ combustion process under consideration would not be expected to introduce any major problems in the subsequent upgrading process.

Résumé Exécutif

PARTIE I:

Les échantillons de pétrole collectionés sur une période d'un an et provenant du puits EX-32 du site de combustion pilote "in situ" de Wolf Lake, en Alberta, furent caractérisés en détail sur les plans physique et chimique. Les propriétés de ces échantillons furent comparées à celles d'un échantillon de carottage (échantillon-témoin) obtenu avant le commencement de l'onde de feu (fireflood). L'objectif de ce travail consistait à évaluer les effets du procédé d'extraction in situ sur la qualité du pétrole produit; une attention particulière fut portée à l'étape suivante du procédé d'enrichissement.

L'enrichissement du pétrole provenant du gisement pilote de combustion in situ était reflété par la viscosité et la densité réduites du pétrole brut et par l'augmentation des quantités de matériel à point d'ébullition dans la région du naphte et dans la région du distillat intermédiaire. Une diminution concomitante du % en poids des fractions de résidu était observée. L'absence d'augmentation du contenu en hétéroatomes ou en asphaltènes durant la portion du cycle de production étudiée était un signe favorable pour l'étape suivante d'enrichissement du pétrole produit.

La fraction de naphte a exhibé des augmentations significatives du contenu en aromatique tôt dans le cycle de production. En plus, il y avait un changement soutenu sous forme de plus petites chaînes carbonées des différentes classes de composés. Cette présence de "nouveaux" composés dans la fraction de naphte résultant du procédé de combustion était soulignée par l'apparition de n-paraffines en C_6 à C_{12}.

Le contenu en aromatique de la fraction du distillat intermédiaire a aussi augmenté tôt dans le cycle de production bien qu'il y ait peu d'indication de changements permanents des classes de composés ou de la distribution du nombre de carbones. C'était la croissance relative en % du poids de la fraction du distillat intermédiaire qui a le plus affecté la qualité du pétrole produit.

Le % en poids des fractions de gas-oil a subit peu de changement résultant de l'onde de feu et il a semblé qu'il y avait peu de changement en rapport avec les classes de composés dans les fractions chromatographiques. Il y eut, toutefois, des changements sous forme d'un plus petit nombre de carbones pour certaines de ces classes.

Le % en poids des fractions de résidu du pétrole produit par l'onde de feu était plus petit que celui des fractions correspondantes provenant de l'échantillon-témoin. Il n'y avait pas de changement significatif du contenu en azote ou en soufre du résidu au cours de cette étude. En plus, il y avait des augmentations négligeables des composantes asphaltènes et polaires des fractions de résidu provenant du pétrole produit relativement à l'échantillon-témoin.

Il est généralement conclu que la qualité du pétrole produit à EX-32 durant la période d'un an couvrant ce procédé de combustion d'oxygène in situ ne devrait pas causer de problèmes majeurs à l'étape suivante d'enrichissement du pétrole. Au contraire, les propriétés du pétrole suggèrent que le processus même de récupération a résulté en un degré d'enrichissement petit mais significatif.

PARTIE II:

Les échantillons de pétrole collectionnés sur une période de huit mois et provenant du puits EX-6, du site de combustion pilote in situ de Wolf Lake, en Alberta, furent caractérisés en détail sur les plans physique et chimique. Les propriétés de ces échantillons furent comparées à la fois à celles du pétrole provenant d'un échantillon de carottage obtenu avant le commencement de l'onde de feu et à celles du pétrole provenant du puits EX-32. L'objectif de ce travail consistait à évaluer les effets du procédé d'extraction in situ sur la qualité du pétrole produit; une attention particulière fut portée à l'étape suivante du procédé d'enrichissement.

Plusieurs techniques analytiques furent appliquées au pétrole brut dont quatre échantillons furent choisis pour mise en fractions par distillation et par chromatographie en colonne. Ces fractions furent caractérisées en détail par chromatographie en phase gazeuse et par spectrométrie de masse (CG-SM).

Par rapport à l'échantillon-témoin, le pétrole brut de EX-6 produit au cours du procédé de combustion in situ a présenté une diminution en viscosité qui a été corrélée à une augmentation de la quantité de matériel à point d'ébullition dans la région du naphte. Contrairement au pétrole de EX-32, le pétrole de EX-6 n'a pas montré d'augmentation de la fraction du distillat intermédiaire.

Les analyses d'éléments et le nombre d'acides du pétrole de EX-6 ont indiqué que les effets chimiques, dûs à toute oxidation à basse température qui a résultée du procédé de combustion in situ, étaient minimes. Basé sur les résultats analytiques, il n'y a pas de fondement chimique

(tel qu'une augmentation de surfactants basés sur les hydrocarbones) expliquant les émulsions "stables" que ces pétroles produits par onde de feu forment.

Les analyses de CG-SM des coupes de distillation du naphte et de la fraction saturée des coupes de distillat intermédiaire du pétrole de EX-6 ont indiqué peu de changements significatifs soit dans la classe de composés ou dans la distribution du nombre de carbones. En contraste avec les résultats du pétrole provenant de EX-32, il n'y avait pas d'évidence chimique suggérant toute dilution (du pétrole produit à EX-6) par des matériaux légers soit distillés près de la ligne de feu, soit produits par craquage thermique à la ligne de feu. Globalement, le procédé de combustion in situ a causé seulement des changements chimiques minimes du pétrole produit à EX-6.

En général, le pétrole produit à EX-6 fut enrichi relativement à l'échantillon de carottage mais à un degré moindre que le pétrole produit à EX-32. On ne s'attend pas à ce que la qualité du pétrole, produit à EX-6 durant la période du procédé de combustion in situ sous considération, introduise des problèmes majeurs à l'étape suivante du procédé d'enrichissement.

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PART I

INTRODUCTION

As conventional oil deposits continue to decline worldwide, there has been an increasing emphasis on the use of heavy oil and bitumen. The high viscosity of these materials, however, results in little or no primary production from their deposits. The application of heat is usually required to reduce the viscosity of the oil and allow it to flow.

A variety of processes have been developed for in situ bitumen recovery. Most supply heat in the form of steam or in situ combustion to reduce viscosity and mobilize the oil in the formation. The application of heat often produces more than physical changes in the oil. In particular, the relatively high temperatures to which the reservoir oil is subjected during in situ combustion can cause chemical changes as well. A number of studies have shown that under these conditions, thermal cracking, low temperature oxidation, coke formation, and other chemical changes can take place [1-10]. These chemical changes clearly have consequences with respect to production of the oil. In addition, the chemical composition and quality of the produced oil are major determinants in the upgrading process.

The recovery of heavy oil and bitumen by in situ combustion (or fireflood) has been receiving increasing attention in recent years. The results of detailed analyses on several samples produced by an in situ combustion process in a heavy oil reservoir in Saskatchewan, Canada were compared to those of oil produced from the same field under

primary recovery conditions [1,2]. A large increase in the naphtha distillation fraction was observed as was a marked increase in the aromaticity of the lower boiling fractions.

The present study is concerned with an in situ combustion field pilot at an oil sands reservoir at Wolf Lake, Alberta. Under a previous contract involving this reservoir, a comprehensive comparison of properties between oil extracted from a core sample and oil subsequently produced by in situ oxygen combustion over a three month period was completed. The main objective of this work was to assess the changes in quality of the in situ combustion-produced oil with respect to the subsequent upgrading process. A draft report describing this work (hereafter called phase one) was prepared and submitted [11]. The results of this study revealed significant differences between the oil produced by fireflood and that derived from the core sample. Large reductions in viscosity and density for production samples were accompanied by substantial increases in the percentages of materials boiling in the naphtha and middle distillate ranges. The produced oil exhibited these effects very early in the production cycle. It was concluded that the quality of this oil would cause no special problems in subsequent upgrading processes.

A determination of the oil quality produced during later stages of the in situ oxygen combustion process was of crucial importance in establishing the long term utility of this recovery process. A second phase of this study was undertaken, covering the period from seven to eleven months after initiation of the fireflood. The results from phase two are featured in this report, although some pertinent phase one data is also included to highlight long term trends in the properties of the produced oil. These data collectively detail the effects of the in situ combustion process on the properties of the produced oil over a one year period.

EXPERIMENTAL

All production samples were collected from an in fill drilled well (EX32) in a five spot pattern. These samples were labelled with the dates they were collected (year/month/day e.g. 880327); the fireflood commenced on approximately September 1, 1987. Previous to this, a core sample was obtained from the same field. The core sample was prepared by toluene extraction and may have lost some light ends during subsequent rotary evaporation, despite the fact that it contained 0.5% residual toluene. Each production sample was dewatered by ultracentrifugation prior to analysis.

Analyses were conducted on the whole oil and subsequent distillation cuts. Each sample was distilled into naphtha (115-204 °C), middle distillate (204-345 °C), gas oil (345-442 °C), and residue (>442 °C) cuts. The latter three cuts were further fractionated by column chromatographic separation. A flow scheme illustrating the separation procedure and the analyses performed on the various fractions is shown in Figure 1.

The density of the whole oil was determined at 25 °C using an Anton Parr Model DMA60 density meter with a DMA601 measuring tube capable of measuring to five decimal places. The instrument was thermostated using a recirculating temperature bath with a control accuracy of ±0.01 °C. The temperature was monitored to the nearest 0.1 °C.

The viscosity measurements were performed on a Brookfield Viscometer model RVT equipped with a cone and plate measuring head. The viscometer was maintained at the measuring temperatures of 25, 60, and 80 °C using a temperature bath capable of

controlling to ±0.2 °C at the viscometer head.

The %water analyses were performed on a Mitsubishi Model CA-02 Karl Fischer instrument.

The nitrogen analyses for the gas oil and residue distillation cuts were determined on a Perkin-Elmer 240 Elemental Analyzer. The low nitrogen content of both the naphtha and middle distillate cuts necessitated the use of a Dohrman Chemiluminescence instrument for the nitrogen analyses. The wt% sulfur in the distillation cuts was determined by the ASTM D1552 (combustion) procedure.

The distillation cuts of the whole oil samples were produced using a Nester Faust Spinning Band Distillation system. This system consists of a 480 mm x 7 mm column with a Monel band spinning at approximately 29 rev/s. The distillations were conducted at a pressure of 5.0 mm Hg and a reflux rate of 5:1.

The aromatic and olefin contents of each naphtha cut were determined using the Fluorescence Indicator method (FIA) ASTM D1319. The aromatic contents of the naphtha, middle distillate, and gas oil cuts were also analyzed by supercritical fluid chromatography (SFC). The SFC determinations were performed using a 250 mm x 2.1 mm I.D. 5μ silica Adsorbosphere column with supercritical CO_2 at 3600 psi as the carrier fluid [12].

The chromatographic type separations (saturates, aromatics, polars, and asphaltenes or SAPA) of the middle distillate, gas oil, and residue distillation cuts were performed using a modification of the method of Sawatzky [13]. After removal of the asphaltenes by pentane

precipitation, the rest of the sample (maltenes) was separated on the column using the following solvent system. Fraction 1 (saturates) was collected with 150 mL of pentane followed by fraction 2 (mainly monoaromatics), which was eluted with 200 mL of pentane containing 5% toluene. Fraction 3 (mostly di- and polyaromatics) was collected with 300 mL of 30% toluene in pentane while fraction 4 (polars) was eluted with 300 mL of toluene/pentane (70/30, v/v). The remaining polar fraction 5 was eluted with 300 mL of methylene chloride/methanol (50/50, v/v). The chromatographic solvents were removed by evaporation for 24 h in a fumehood. Any residual solvent was removed by heating in an oven at 100 °C for 10 min. The total recoveries from the chromatographic separation of the residue cuts were lower than expected (91-99%). This was attributed to varying amounts of a dark, polar material which did not elute from the chromatographic adsorbent.

The ^{1}H and ^{13}C nuclear magnetic resonance (NMR) data were measured on a Bruker WP-80 NMR spectrometer. Samples were measured in $CDCl_3$ solution and referenced to residual $CHCl_3$. The integrated ^{1}H spectra were divided into aliphatic (0.0 to 6.0 ppm) and aromatic (6.0 to 9.3 ppm) regions. The integrated ^{13}C NMR spectra were also divided into aliphatic (0 to 70 ppm) and aromatic (118 to 160 ppm) portions.

The gas chromatography-mass spectrometry (GC-MS) analyses were carried out on at Finnigan Mat 4500 Gas Chromatograph - Mass Spectrometer. Methyl silicone DB-5 or Hewlett Packard P.N.A. capillary columns were used for the gas chromatographic separations. The methyl silicone DB-5 column was 25 m by 0.21 mm I.D. with a 0.33μ film thickness. The P.N.A. column was 30 m by 0.25 mm I.D. with a 0.1μ film thickness. A split injection system was used for sample introduction on both capillary columns. For the DB-5

column, the gas chromatography was performed isothermally at 65 °C for 5 min followed by temperature programming at 10 °C per min up to 295 °C. The helium carrier flow rate was 35 cm per sec measured at 65 °C. For the P.N.A. column, the separations were performed isothermally at 35 °C for 10 min followed by two temperature programs: 3 °C per min to 150 °C then 10 °C per min to 200 °C. The helium carrier flow rate on this column was 27 cm per sec measured at 65 °C.

The mass spectra of the gas chromatographic effluents were obtained using 70 eV electron impact (EI) ionization or nitrous oxide (N_2O) chemical ionization (CI) as specified. The scan rate was generally 0.95 sec to scan from 33 to 500 amu. The CI spectra were obtained using a Townsend discharge ionization source with N_2O at approximately 0.6 torr at 120 °C. The spectra were summed over the length of the chromatogram to give a picture of the types of chemical structures found in these samples. The response of the mass spectrometer detector for different molecular weight components was not determined due to a lack of suitable standard compounds. It is generally recognized that the response drops off with increasing molecular weight and thus the data presented are not an accurate representation of the relative concentrations of compounds within a particular sample. The data do, however, accurately represent the differences between samples and do give a semi-quantitative picture of the distribution of compound types within a given sample.

The residue fraction samples were introduced into the mass spectrometer using a solids probe which was heated from 50 °C to 300 °C at 50 °C per min. The mass spectra were scanned from 150 to 600 amu in 0.95 sec and summed over the entire vaporization time.

The asphaltene agglomeration data for the whole oil samples were measured by an in-house method [14]. Pentane was used to precipitate asphaltenes from a toluene solution of the oil samples. The onset of asphaltene agglomeration was monitored by the increase in absorbance of the solution due to light scattering from the particles.

RESULTS AND DISCUSSION

Whole Oil Analysis

Some analytical data for the whole oils from phase 1 (Samples: Core, 870929, 871023, 871116) and phase 2 are given in Table 1. There is a significant reduction in viscosity between the core sample and the fireflood-produced oils. This observation is consistent with those from previous studies, both in the laboratory and in the field, which involved changes to heavy oils under thermal conditions [1,3,8,10]. It is reasonably clear that this viscosity reduction stems from an increase in the percentages of the naphtha and middle distillate cuts. These distillation data are shown in Table 2. A careful analysis reveals that the viscosity of the produced oil is most closely correlated with the wt% of middle distillate in the sample. The general increase in the wt% of the naphtha and middle distillate cuts for the produced oils versus the core is balanced by a decrease in the weights of the residue cuts.

A consideration of the physical mechanism believed to be operating during the early stages of an in situ combustion process suggests that this viscosity reduction stems from a dilution of the oil with lower boiling material produced and vaporized as a result of thermal cracking near the firefront. It must be appreciated, however, that the recovered oil will consist not only of material formed at the firefront. The steam zone which precedes the combustion region will transfer heat to the virgin or native oil in the formation. The consequent reduction

in viscosity will mobilize this oil and lead to simultaneous production of both virgin oil and volatile material formed near the firefront. The compositional details of the produced oil are perhaps even more complex than this consideration suggests. Laboratory studies have shown that there is fractionation of crude oil as it moves through a quartz matrix, resulting in a redistribution of the saturate hydrocarbon fraction in favour of the lower molecular weight n-alkanes with increasing migration distance [15,16]. This redistribution was accentuated by the presence of clays in the mineral phase. The results from the Saskatchewan study [1,2] indicated that this type of fractionation was occurring during movement of the oil through the reservoir under the influence of the in situ combustion process. Thus, the portion of the produced oil derived from mobilized native material need not be fully representative of the formation oil. Similarly, one might expect the volatile material, formed by thermal cracking at the firefront, to condense and undergo a certain amount of fractionation in the formation. Finally, the fact that the produced oil is from an in fill drilled well leaves open the possibility that oil altered in an earlier fireflood process may be mixed with virgin oil. These considerations must be kept in mind when analyzing the data from the whole oil.

The phase one data (presented in the earlier report [11]) showed that the wt% of both nitrogen and sulfur in the produced oil of phase one decreased slightly relative to the core sample. This reduced heteroatom content in the produced oil samples is clearly an advantage in any subsequent upgrading process. On the other hand, the wt% of oxygen in the produced oil showed a small increase relative to the core sample. However, the produced oil exhibited lower acid numbers, indicating that this oxygen was in a non-polar (i.e. nonacidic) form. Similar results have been observed in related studies involving laboratory simulation of in situ combustion [8]. If any polar oxygen-containing compounds

(e.g. carboxylic acids) are formed during the combustion process, adsorption onto the formation matrix or preferential distribution into the aqueous phase would account for their low concentration in the produced oil.

The asphaltene agglomeration data for the phase two whole oil samples are shown in Table 3. This type of analysis has been used [14] to examine property changes occurring in the oil during thermal recovery processes. The y-intercept is a measure of the propensity for asphaltene precipitation, with smaller numbers indicating a greater tendency for precipitation. Oil affected by thermal recovery processes usually shows an increased tendency for asphaltene precipitation, which may be explained by changes in the maltene fraction of the oil (e.g. lower aromatic content). These changes result in a decreased ability of the whole oil to maintain the asphaltenes in a dissolved state. However, the data in Table 3 do not appear to show any obvious or simple correlation with the variation in the composition or properties of the maltene fractions. The negative y-intercepts for samples 880424 and 880522 may suggest that asphaltene precipitation had already occurred. We are presently unable to offer a rational explanation for these data as a whole.

Given the number of factors affecting the composition of the produced oil, little more can be gained by analyzing the whole oil samples. An analysis of the distillation cuts and chromatographic fractions follows.

Naphtha

The data for the naphtha distillation cuts are shown in Table 4. The naphtha content in each

of the produced oil samples is higher than that of the core sample. In an effort to minimize loss of light ends, some of the toluene used to extract the core sample was left in the naphtha cut. For this reason, a full analysis of this sample was not performed.

The aromatic contents of the samples were measured by supercritical fluid chromatography (SFC) while ASTM D1319 (fluorescent indicator adsorption) was used to measure the aromatic and olefinic components of the samples. These results are shown in Table 4. The balance of the samples by both techniques are saturates, with the olefins included in the saturates by the SFC technique. The results of these two methods are in good agreement when converted to the same units. Early in the production cycle, there is an increase in the amount of olefins present. These are likely formed by thermal cracking near the firefront and produced early in the cycle due to their relatively high volatility. A similar argument may be used to rationalize the increase in aromatics during the early recovery phase. As the production cycle continues, the amounts of both olefins and aromatics decrease in the naphtha fractions. Two plausible explanations for these observations may be advanced. An argument based on the cumulative effects of oil movement and fractionation as the firefront advances is consistent with this data. While the volatile material from the current firefront continues to migrate preferentially, the heat-induced advance (and subsequent fractionation) of the virgin oil is occurring. The composition of the produced oil should eventually reach a steady state with respect to the various compounds produced by thermal cracking and the mobilized native oil. Alternately, the data could be indicating a change in field operating conditions from fireflood to pressure blow-down, thus halting the production of small, volatile compounds by thermal cracking.

A consideration of production dynamics and formation properties suggests that some variation in oil constitution is to be expected as production continues. The irregular advance of the combustion zone, variation in the efficiency of combustion, differences in oil density in the matrix, and channeling effects should lead to fluctuations in the composition of the produced oil. This type of behaviour may be observed in the phase two data with respect to the amounts of olefins and aromatics present.

The amounts of both sulfur and nitrogen in the naphtha cuts of the produced oil show a marked increase relative to the core sample. This is likely due to small, volatile nitrogen- and sulfur-containing molecules formed during the combustion process and produced early in the recovery cycle. The magnitude of the increase in the amount of nitrogen for the 871116 sample is particularly striking. The levels of both sulfur and nitrogen fall off and vary within a limited range in later samples, consistent with the previous arguments based on steady state production dynamics and fractionation.

A GC-MS analysis revealed some changes in the oil as a result of the in situ combustion process. The total ion chromatograms (derived from the GC-MS analysis) for the naphtha cuts of the core and phase one samples are shown in Figure 2. The shift towards peaks of shorter retention times (ie. lower boiling material) as the combustion process proceeds is consistent with the produced oil being diluted with smaller molecules produced near the firefront. The reconstructed ion chromatograms for the straight and branched chain paraffins (or open chain saturates) in the naphtha cuts, shown in Figure 3, reveal further evidence in support of this view. These show that the core sample contains only small amounts of n-paraffins while the phase one samples have increasing amounts of C_6 to C_{12} n-alkanes.

These molecules are likely formed from the scission of alkyl-substituted aromatic compounds or the cleavage of naphthenic rings.

The results of a GC-MS compound type analysis are shown in Figure 4. The core sample consists mainly of alkyl benzenes plus 1- and 2-ring saturates (naphthenes). The samples from early in the combustion process show a relative growth with time of alkyl benzenes, benzocycloparaffins, and the single compound naphthalene. In addition, the distributions of 1- and 2-ring saturates are shifted to lower carbon numbers in the early phases of the fireflood recovery. These changes may also be attributed to thermal cracking of larger molecules at the firefront. There is also a small relative increase in the open chain alkanes during the early phase of the combustion process, consistent with the observed appearance of the homologous n-alkanes in the reconstructed ion chromatograms (Figure 3).

The phase two data reveal more complex changes occurring in the naphtha cuts. The earlier trend for the 1- and 2-ring naphthenes is reversed, with these compound classes now growing relative to the alkyl benzenes. The cumulative effects of fractionation in the formation and the advancing firefront could be responsible for this behaviour, as could a change in the operating conditions of the fireflood process.

An alternate presentation of the GC-MS data is illustrated in Figures 5 and 6. These highlight the changes in carbon number distribution for the open chain saturates and alkyl benzenes, respectively, as a function of time. Both compound classes exhibit similar behaviour as the production cycle progresses: there is a small but persistent shift towards lower carbon number. This result is consistent with the preferential migration of lighter

material through the formation to the production well. While it is tempting to place interpretations on the relative areas under the bargraphs in Figures 5 and 6, these comparisons may not be completely justified. The bargraphs for the individual samples were not normalized to the same constant and small relative changes in areas cannot be considered meaningful. However, the gross reduction in area for both the open chain saturates and alkyl benzenes for the 880327 sample relative to the 871116 sample does appear to be indicative of a change in production conditions. A shift from fireflood to pressure blow-down would be expected to reduce the relative amounts of small, volatile species (such as open chain saturates and alkyl benzenes) in the naphtha portion of the produced oils as thermal cracking at the firefront ceased.

Two general conclusions may be drawn from the previously analyzed data for the naphtha cut. Changes in the relative distribution of the compound classes which occur early in the production cycle tend to be transitory, as witnessed by the behaviour of the 1- and 2-ring naphthenes. On the other hand, the shifts towards lower carbon number in the individual compound classes as the combustion process evolves tend to be more enduring.

Middle Distillate

The data in Table 5 show the large relative increase in the wt% of the middle distillate cuts in the phase one fireflood samples compared to the core sample. As discussed previously, the reduction in viscosity of the fireflood whole oil samples is most closely correlated with the wt% of this cut. The amount of nitrogen increases dramatically during phase one but returns to lower levels late in phase two of the recovery process. The amount of sulfur

follows a similar but less striking variation to that of nitrogen. These observations are consistent with previous arguments based on the formation of small nitrogen- and sulfur-containing molecules which are produced early in the production cycle.

The aromatic contents of the middle distillate cuts show an increase relative to the core material for the phase one samples but return to core levels later in phase two. Similar behaviour was observed for the wt% of aromatics in the naphtha cuts and a similar explanation may be advanced. The early enrichment of the produced oil by volatile aromatic molecules produced by thermal cracking is later moderated by the increasing production of mobilized and fractionated virgin oil.

The chromatographic yields for the middle distillate cuts are shown in Table 6. The increase in wt% of aromatics early in the combustion cycle (by SFC) is reflected in the increased wt% of fraction 3 for the 871023 and 871116 samples. The variation in wt% aromatics is inversely related to the wt% of saturates, the polar fractions 4 and 5 varying little over the production cycle.

A set of GC-MS compound type analyses were performed on the chromatographic fractions from the middle distillate cuts. Figures 7 and 8 show the distribution of compound classes for fractions 1 and 2, respectively. Fraction 1 (saturates) is composed mainly of 1 to 3 ring naphthenes and shows little change in compound class distribution over the length of the production cycle. The major components of fraction 2 (aromatics) are alkyl benzenes and benzocycloparaffins. Again, there are no significant trends in compound class distribution over time, although the alkyl benzenes do show a relative increase for the 871116 and

880522 samples.

The behaviour of the carbon number distribution for fractions 1 and 2 is similar to that observed for the compound class distribution. The open chain saturates from fraction 1 and the alkyl benzenes from fraction 2, both as a function of time, are shown in Figures 9 and 10. It may be observed that the distribution of carbon numbers changes little for either group as the in situ combustion process proceeds.

Chromatographic fraction 3, which contains the bulk of the di- and polyaromatics, exhibits some variation in the relative distribution of compound types. As may be observed in Figure 11, the 871023 sample shows dramatic increases in the single compounds naphthalene and phenanthrene/anthracene. While this increase is short-lived for the former, the enhancement of the unsubstituted triaromatics persists into phase two. The large relative value for the wt% of fraction 3 in the 871023 sample (Table 6) can be associated with these increases in naphthalene and phenanthrene/anthracene. It may also be noted (Figure 11) that this latter compound type, as a whole, exhibits a slow but steady growth in relative intensity during the time period of the study.

The alkyl benzenes in fraction 3 show some variation in carbon number as the fireflood proceeds. There is a shift towards lower carbon number later in phase one which reverses itself as phase two progresses. The variation in relative intensity for the benzocyclo- and benzodicycloparaffins appears to be complex. While some of the changes taking place in fraction 3 may be related to dilution of the oil with fractionated products formed near the firefront, the details are not clear.

The GC-MS analysis of the distribution of compound classes in fraction 4 of the middle distillate cuts is shown in Figure 12. The dramatic shifts in compound type for the core and phase one samples is more apparent than real, as the core and 871023 data are for fractions 4 and 5 combined. Even taking this fact into account, the relative changes between compound classes for fraction 4 are of interest. As the in situ combustion process occurs, there is an increase in the phenanthrene/anthracene content with a concomitant decrease in alkyl benzenes and benzocycloparaffins. These changes could be a result of new compound formation via condensation type reactions, as opposed to thermal cracking. The oxidation of alkyl benzene or benzocycloparaffin side chains (a plausible occurrence during in situ oxygen combustion) could provide the necessary substrates to synthesize phenanthrenes and anthracenes by ring closure [17].

Fraction 4 contains a high proportion of dibenzothiophenes and a two-ring terpenoid sulfide series. These two classes were measured together as their molecular weights are the same, but a fragmentation ion study revealed that both series were present. Figure 13 shows the changes to this compound class as a function of time. Again, there is a shift to lower carbon number early in the production cycle which is reversed during the later phases. This behaviour was common to almost all of the compound classes in this fraction and is consistent with earlier arguments. Fractionation of the mobilized native oil in the formation would result in a lower carbon number distribution in the produced oil relative to the core sample during early phases of the combustion process. This shift would be reversed as the heavier (i.e. more "retained") components in each compound class were produced later in the cycle. The polar nature of the compound classes in this fraction argues against the presence of any firefront-affected material.

Some ^{1}H and ^{13}C nuclear magnetic resonance (NMR) data for fractions 3, 4, and 5 of the middle distillate cuts are shown in Table 7. In the case of the ^{1}H NMR data, the spectrum is divided into the number percent of hydrogens attached to aliphatic structures (i.e. alkyl chains and naphthenic rings) and the analogous percent of hydrogens attached directly to aromatic (e.g. benzene, naphthalene, etc.) rings. For the ^{13}C NMR data, the corresponding division is between carbon atoms contained in aliphatic or naphthenic structures and those carbon atoms contained in aromatic rings. The %aromatic carbon or hydrogen is often referred to as the aromaticity. The data in Table 7 reveal that the carbon aromaticity is always greater than the hydrogen aromaticity. This is the expected result, in that condensed di- and polyaromatic rings contain some aromatic carbon atoms which lack attached hydrogen atoms. Also, there are on average two aliphatic hydrogens for every one aliphatic carbon.

The changes in the NMR data in Table 7 appear to correlate with variations in the compound class distribution for the chromatographic fractions. For example, the increase in aromaticity for fraction 3 early in phase one is consistent with the dramatic increase in the amounts of naphthalene and phenanthrene/anthracene during this period (Figure 11). The small sample size available for some of the fraction 4 and 5 samples resulted in large uncertainties in the aromaticity due to noisy spectra. These uncertainties make meaningful analysis of the NMR data for these fractions difficult.

Gas Oil

Some data for the gas oil distillation cuts are shown in Table 8. There is no clear trend to

the variations in wt% for this cut. These variations are likely due to factors such as channeling and formation heterogeneities. There is little change in either the nitrogen or sulfur content between the core and produced oil samples. The aromatics content of the core and 870929 samples are low but increase dramatically to an approximately constant level for the 871023 and subsequent samples. This behaviour does not correlate with the NMR data (see below) or the GC-MS compound class analyses and we presently have no adequate explanation for this result. The column chromatographic data for the gas oil cuts are shown in Table 9. There are few obvious trends in the fraction yields. Most of the major changes involve the redistribution of aromatics between fractions 2 and 3.

GC-MS analyses for fractions 1, 2, and 3 of the gas oil distillation cuts were performed. The compound class distribution for the saturates fraction 1 (Figure 14) reveals some small shifts such as a transitory relative increase in the open chain compounds for a few of the samples and an increase in the 3- and 4-ring naphthenes later in the process. The GC-MS group type analysis for the aromatics fraction 2 (Figure 15) shows a slow but consistent shift from mono- to diaromatics as the production cycle progresses, as shown by the relative increase in groups 4, 5, 6, and 7. Figure 16 shows the GC-MS analysis of chromatographic fraction 3 from the gas oil distillation cuts. The most notable change is the relative increase in the naphthalene and phenanthrene/anthracene compound classes during the fireflood process. In general, however, the GC-MS analyses of the gas oil fractions reveal only small changes in the distributions between compound classes.

There are changes with respect to carbon number distribution in some of the gas oil chromatographic fractions. The open chain group from the saturates fraction 1 and the alkyl

benzenes from fraction 2 are shown as a function of time in Figures 17 and 18, respectively. A slow but sustained shift towards lower carbon number is observed for both compound classes over the period of the study, in accord with fractionation of the oil during migration through the formation. A shift toward higher carbon numbers might be expected for samples collected from this well subsequent to the period of this study.

The ^{1}H and ^{13}C NMR data for chromatographic fractions 3 and 5 (fraction 4 not measured) of the gas oil distillation cuts are shown in Table 10. Overall, the aromaticities are variable and no consistent trends are evident. There are no obvious correlations between the fraction 3 NMR data and the GC-MS compound class analysis for this fraction (Figure 16).

Residue

Some data for the residue cuts of the core and fireflood-produced oils are given in Table 11. The wt% of the residue cuts decrease significantly during phase one. While there is a reversal of this trend for the phase two samples, a return to the core sample levels does not occur. This "trough" in the wt% of the residue cut correlates with the increased wt% of the middle distillate cut during this period (Table 2). There is no significant change in the wt% of nitrogen or sulfur over the time period of the study.

The column chromatographic yields for the residue cuts are shown in Table 12. Of considerable interest is the negligible increase in the asphaltene content of the produced oils relative to that of the core sample. Any low temperature oxidation occurring during the in situ combustion process is clearly not increasing the asphaltene content of the produced oil.

One would again expect no major problems in the subsequent upgrading process. Similarly, no general increase in polar material is observed in the residue cuts. It may be observed that the wt% of fraction 3 (di- and polyaromatics) undergoes a gradual decrease as the in situ combustion process proceeds.

The yields for all of the chromatographic fractions undergo considerable fluctuations during the period of the study. The dynamics of the formation and the in situ process are likely causes of this behaviour. Changes in the conformance and temperature of the combustion zone coupled with formation heterogeneity would be expected to cause substantial variations in the composition of the produced oil. The low but variable column recoveries (see Experimental Section) might also contribute to these fluctuations.

A set of GC-MS compound class analyses were performed on chromatographic fractions 1, 2, and 3. Figure 19 shows the distribution of compound classes for fraction 1 of the residue cuts. The 4- and 5-ring naphthenes are the predominant compound types for all of the samples, although the open chain, 1-, and 2-ring classes all show a relative increase as the production cycle proceeds. The GC-MS analysis of fraction 2 is shown in Figure 20, where it may be observed that the distribution of compound classes changes little over the time period of the study. The GC-MS data in Figure 21 show the compound type analysis for fraction 3 of the phase two residue cuts. Again, the data reveal only marginal changes occurring during this period of the recovery process. Clearly, the effects of the fireflood recovery process are not manifested in the compound distributions within the residue cuts. It should also be noted that Figures 19, 20, and 21 represent only those portions of the respective fractions with sufficient volatility to be observed by the GC-MS technique.

There are variations in the distribution of carbon numbers within some of the compound classes as the combustion process proceeds. These variations, however, are not uniform. An example of this is depicted in Figure 22, which shows the change in the open chain saturates from fraction 1 as a function of time. The maximum in the carbon number distribution changes several times over the time period of the study. This may again be attributed to the complex nature of the recovery process.

The ^{1}H and ^{13}C NMR data for chromatographic fractions 3 and 5 (fraction 4 not measured) of the residue distillation cuts are shown in Table 13. Overall, the aromaticities show little variation over the period of this study. This result is consistent with the GC-MS compound class analysis for fraction 3 of the residue cuts (Figure 21), which revealed only marginal changes as the production cycle proceeded.

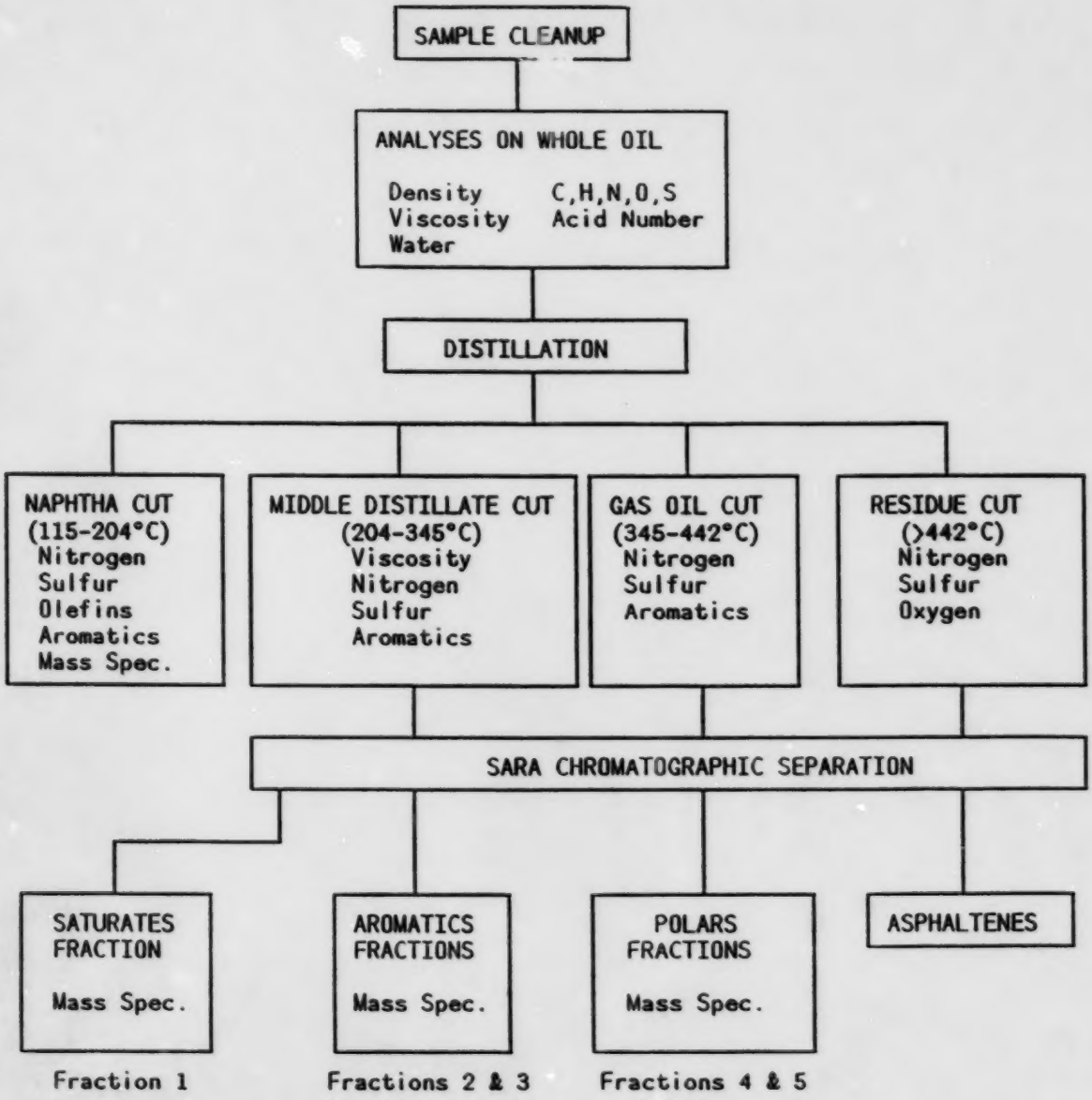


Figure 1: Flow scheme showing sample separation procedure and analytical techniques.

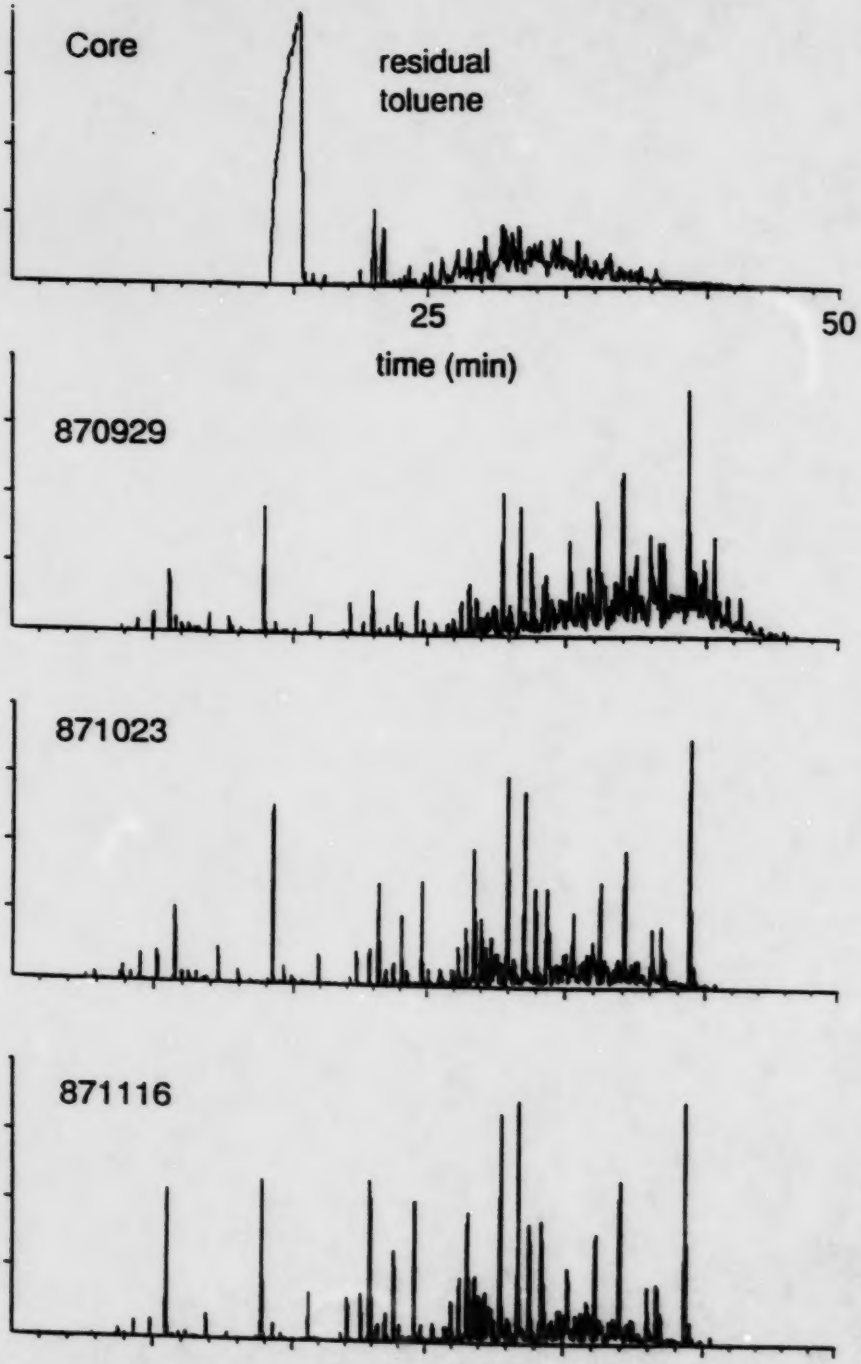


Figure 2. The mass spectrum total ion chromatograms for the naphtha cuts of the core and phase one samples. Ion current in arbitrary units on the y-axis.

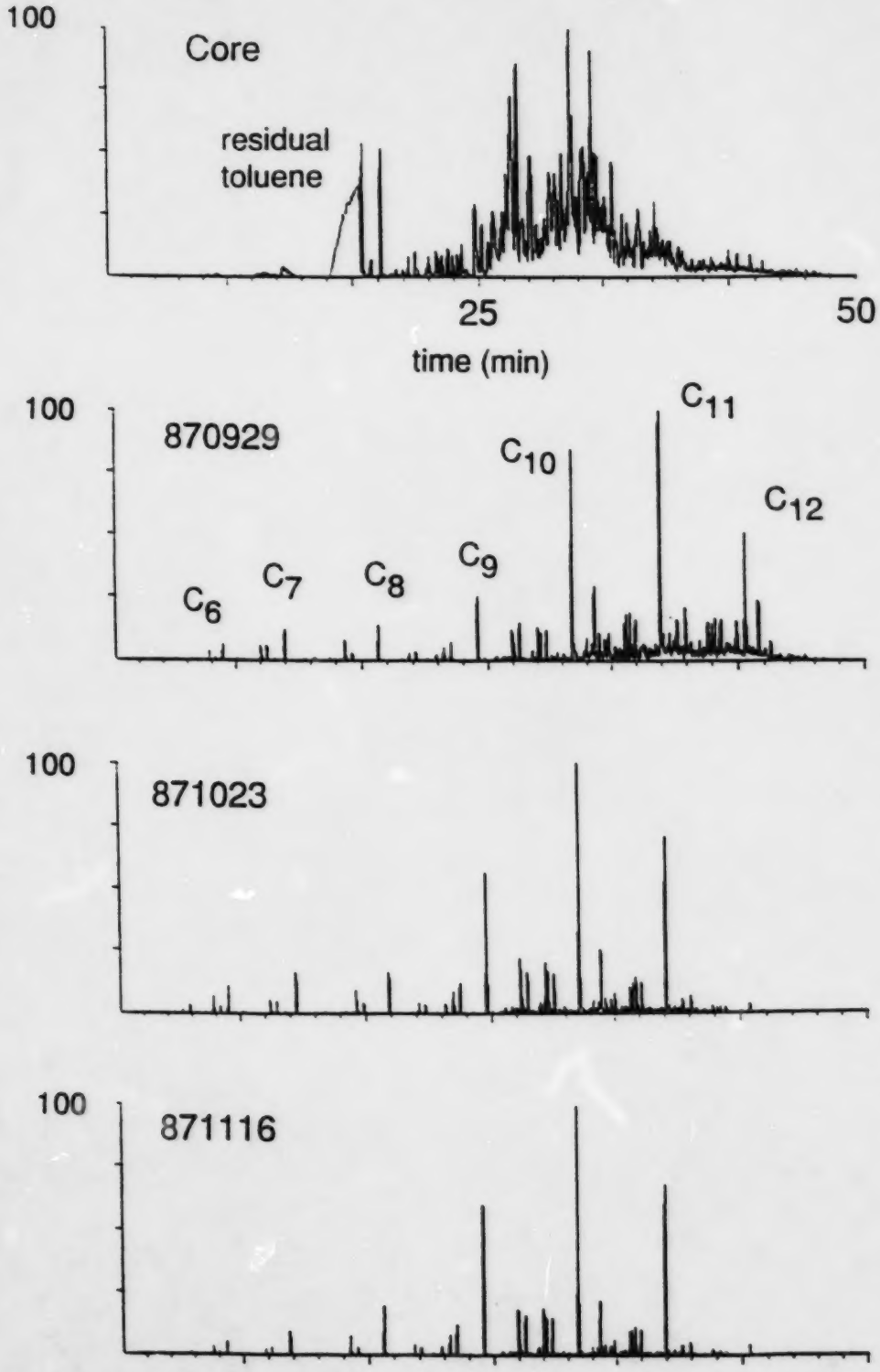


Figure 3. Reconstructed ion chromatograms for the open chain saturates in the naphtha cuts of the core and phase one samples. Relative ion current (%) on the y-axis.

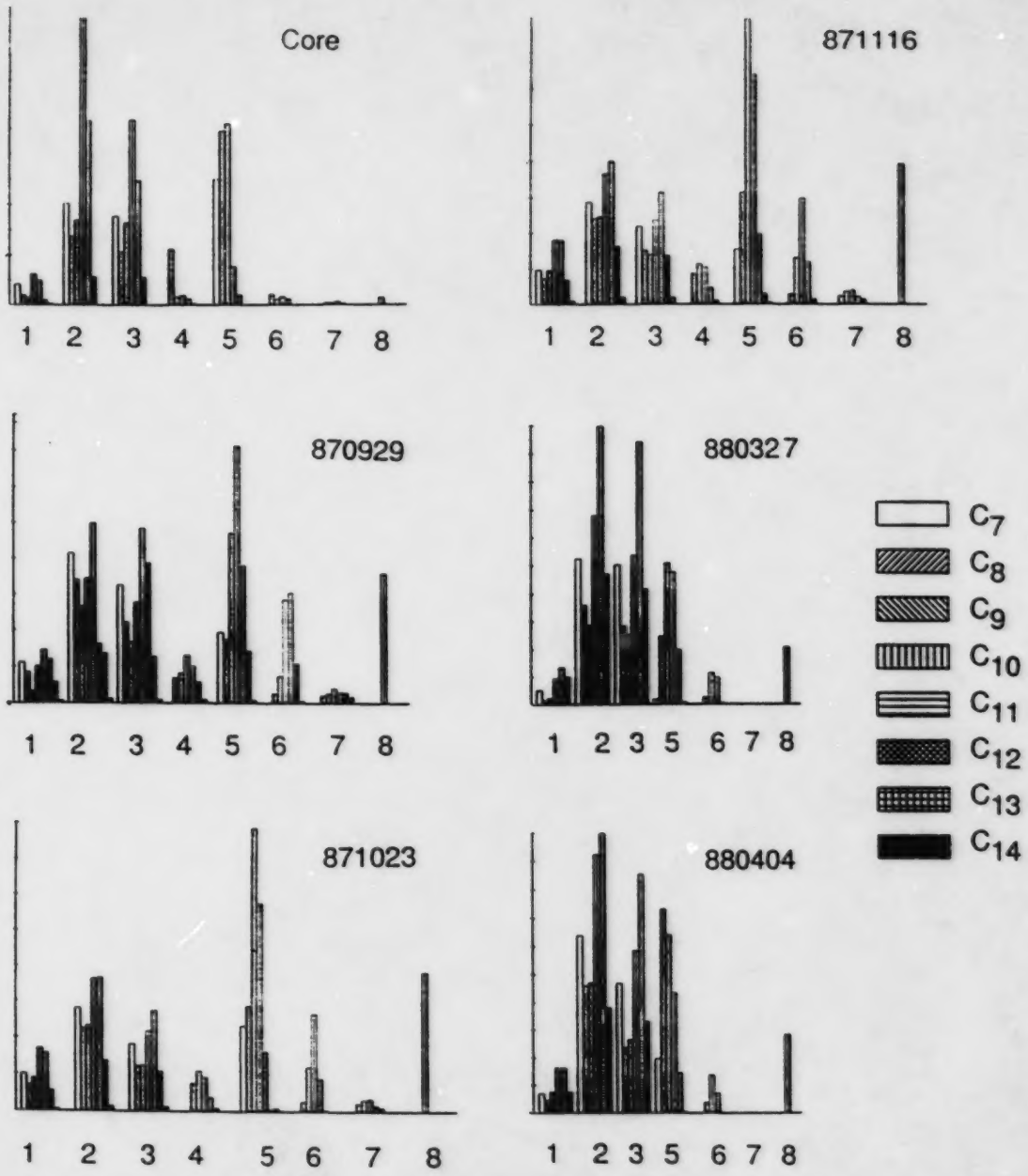


Figure 4. GC-MS analysis of the distribution of compound classes in the naphtha cuts. Intensity (arbitrary units) on y-axis.

1= open chain saturates
2= one-ring saturates
3= two-ring saturates
4= three-ring saturates
5= alkyl benzenes
6= benzocycloparaffins
7= dibenzocycloparaffins
8= naphthalene

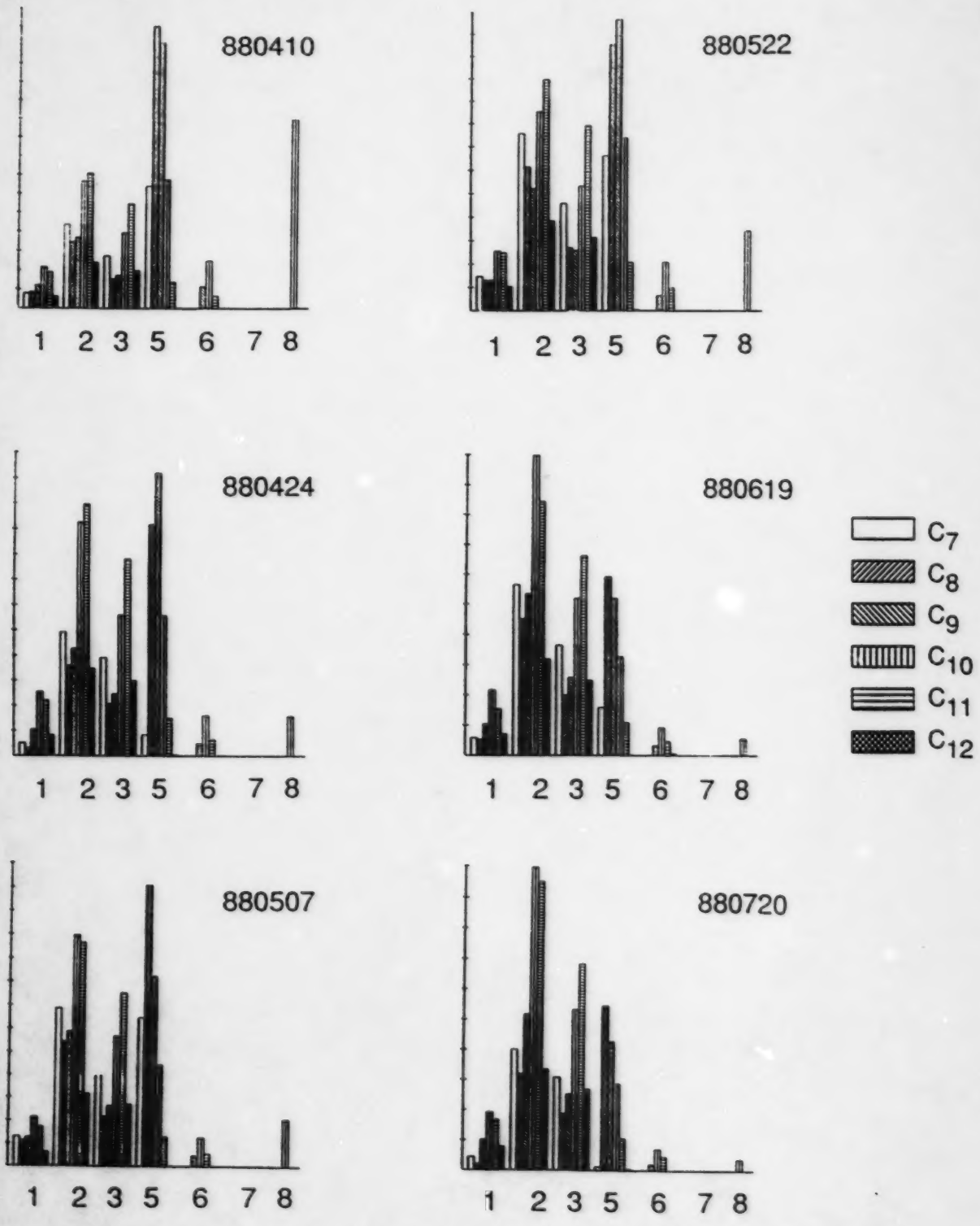


Figure 4. (cont.)

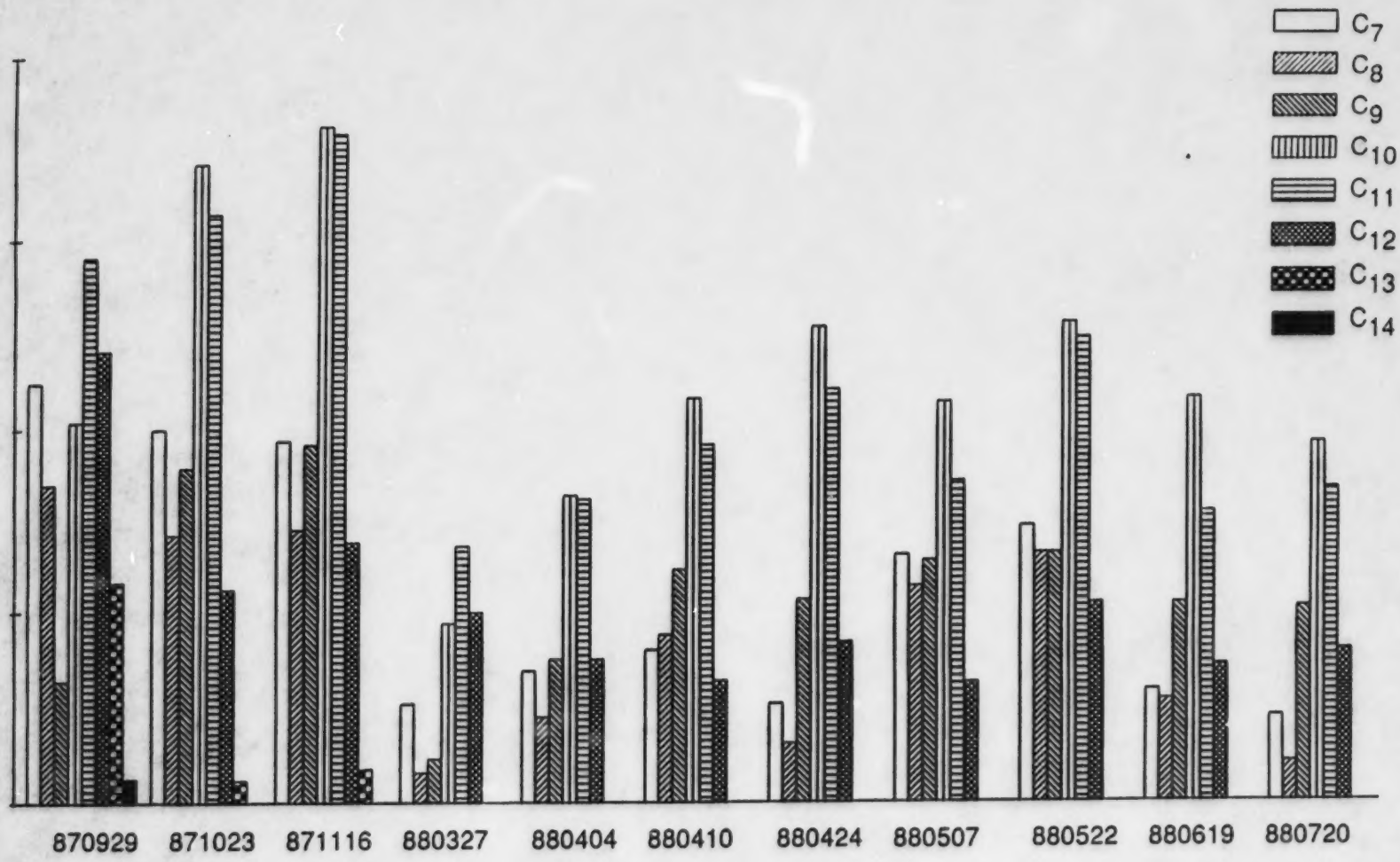


Figure 5. GC-MS analysis of the open chain saturate compound class from the naphtha cuts over the time period of the study.

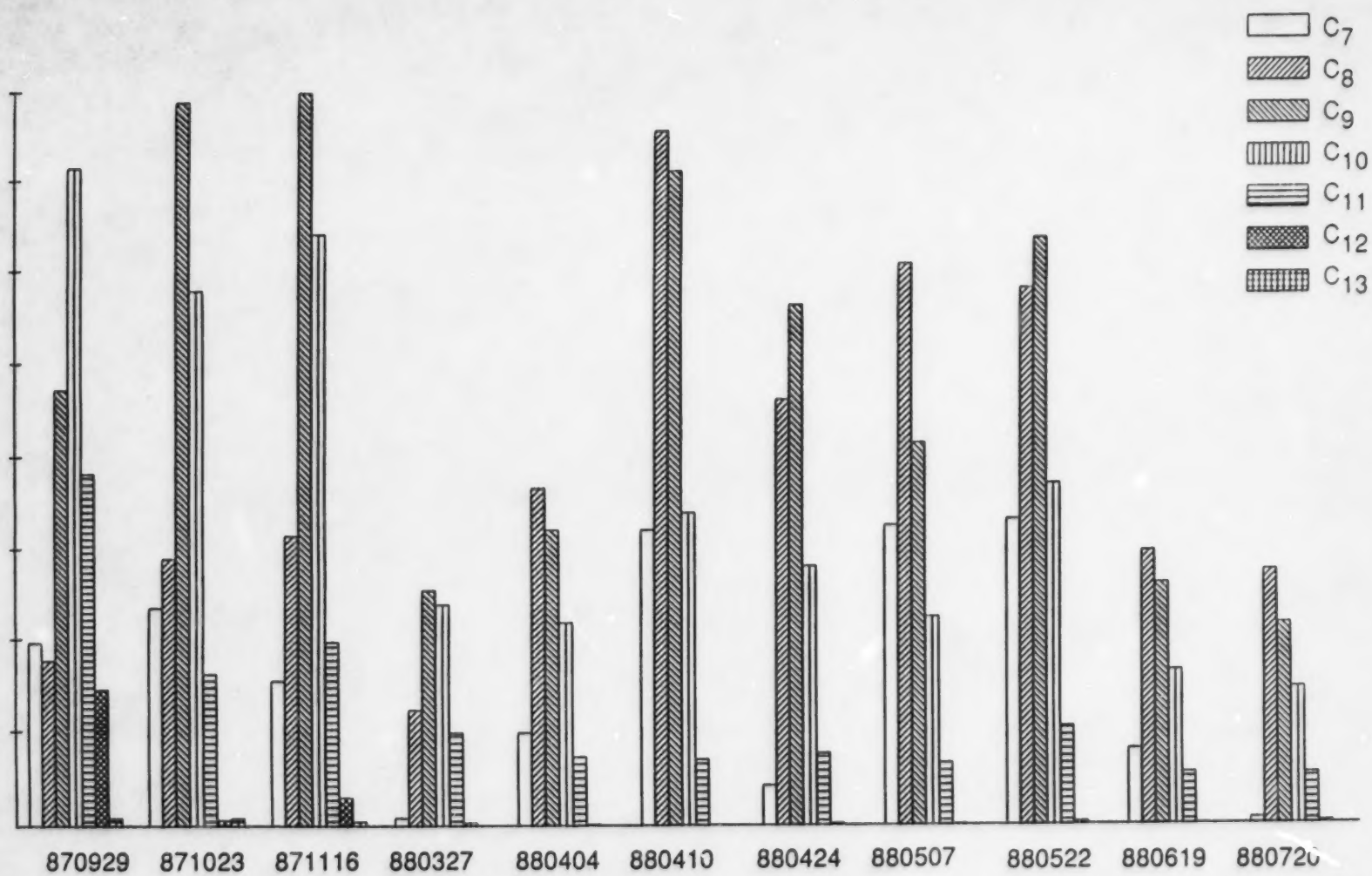


Figure 6. GC-MS analysis of the alkyl benzene compound class from the naphtha cuts over the time period of the study.

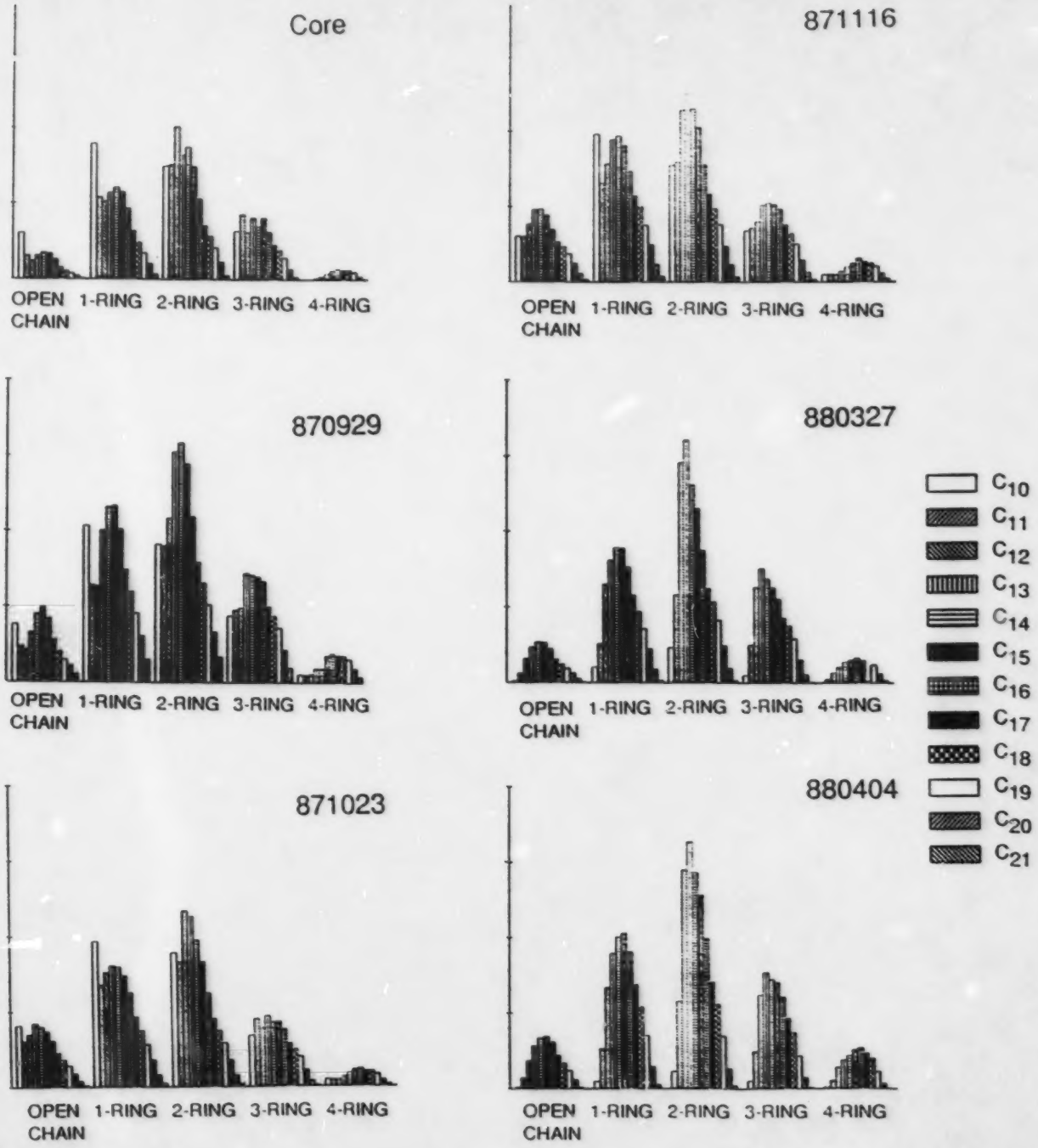


Figure 7. GC-MS analysis of the distribution of compound classes in fraction 1 of the middle distillate cuts.

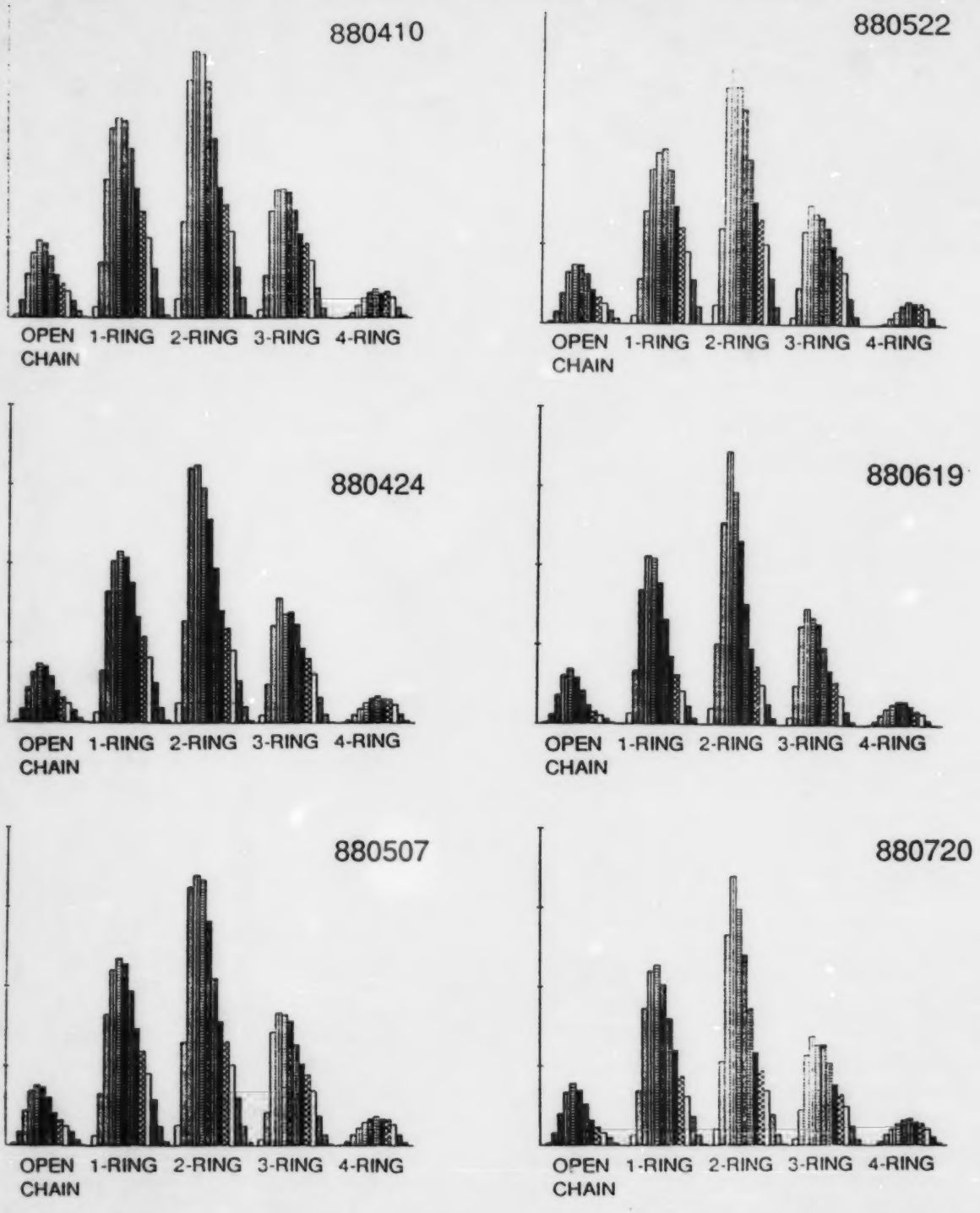


Figure 7. (cont.)

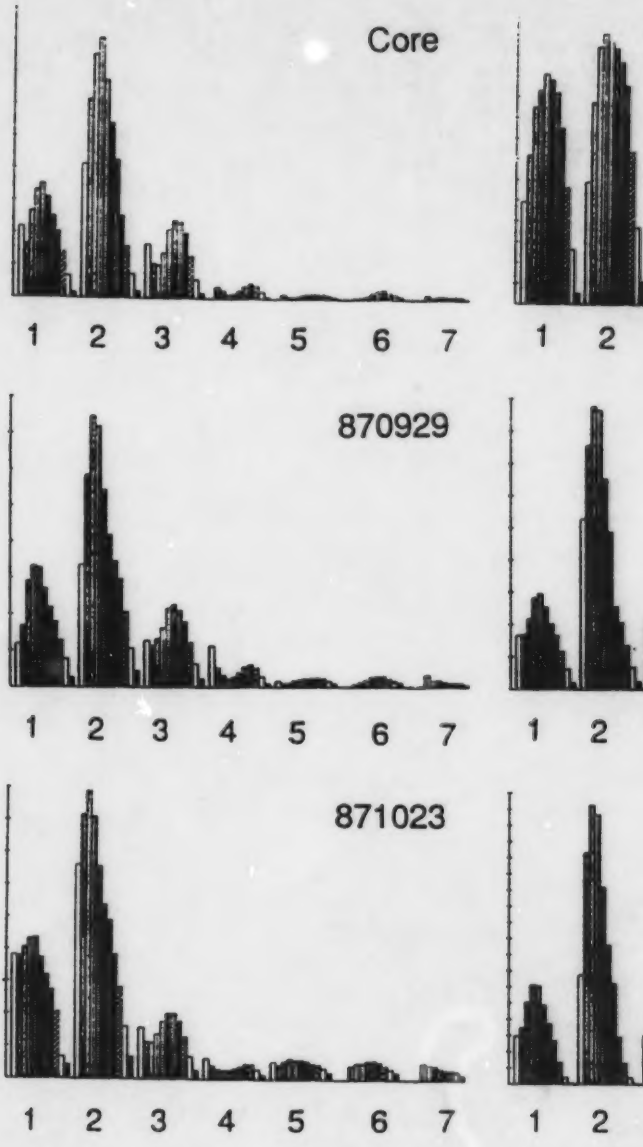
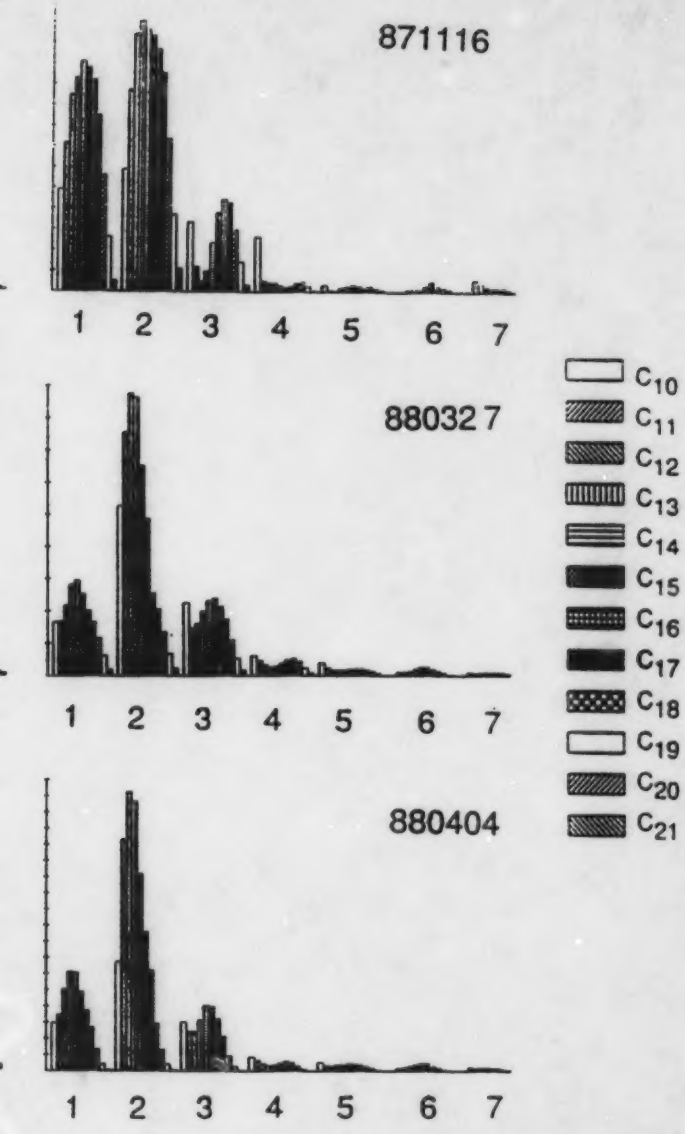


Figure 8. GC-MS analysis of the distribution
fraction 2 of the middle distillate cu

1= alkyl benzenes
2= benzocycloparaffins
3= benzodicycloparaffins
4= naphthalenes
5= naphtho
6= fluorene
7= phenant



e distribution of compound classes in
e distillate cuts.

5= naphthocycloparaffins
6= fluorenes
7= phenanthrenes/anthracenes

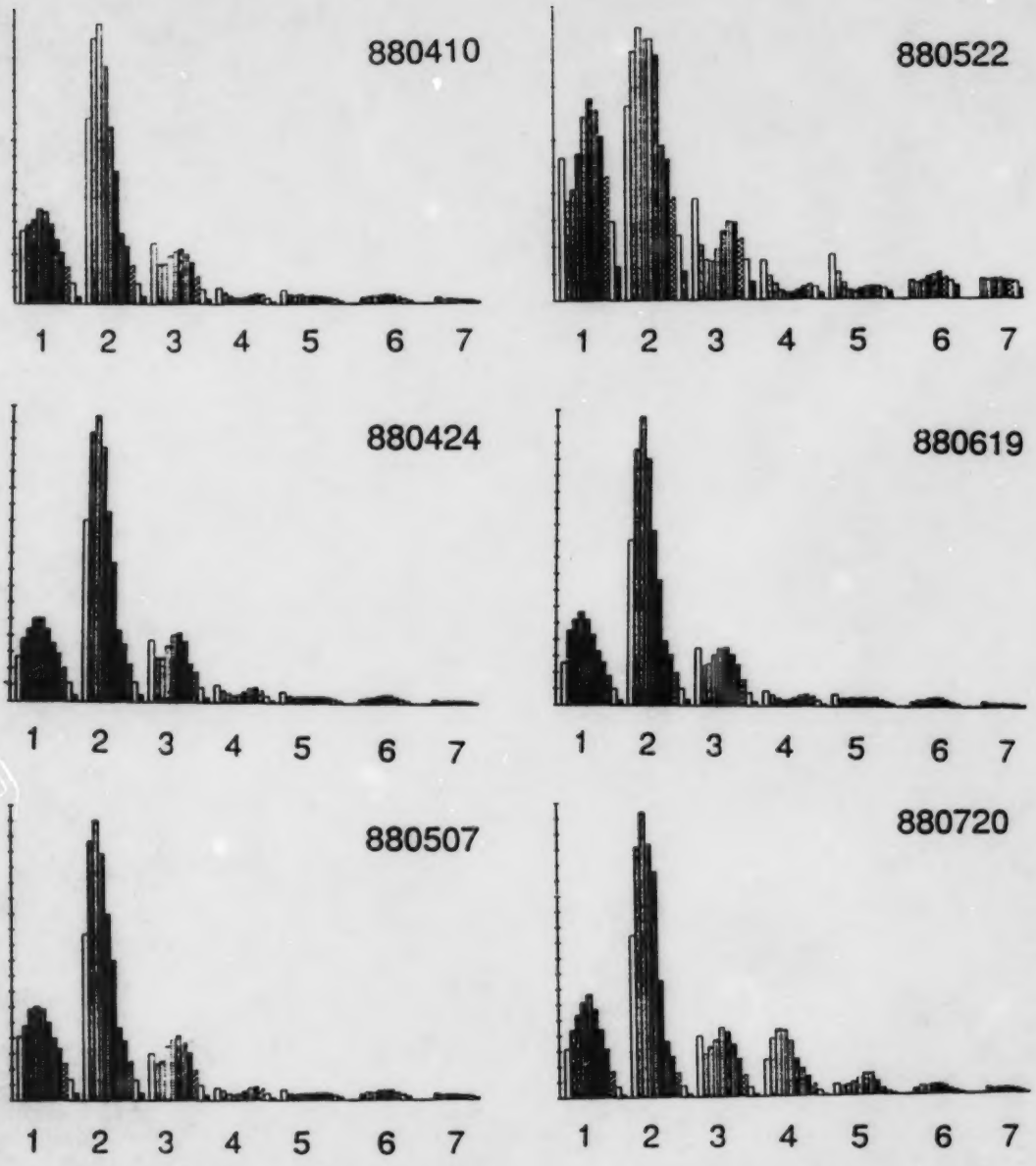


Figure 8. (cont.)

C_{10} C_{11} C_{12} C_{13} C_{14} C_{15} C_{16} C_{17} C_{18} C_{19} C_{20} C_{21}

1= alkyl benzenes
2= benzocycloparaffins
3= benzodicycloparaffins
4= naphthalenes
5= naphthocycloparaffins
6= fluorenes
7= phenanthrenes/ anthracenes

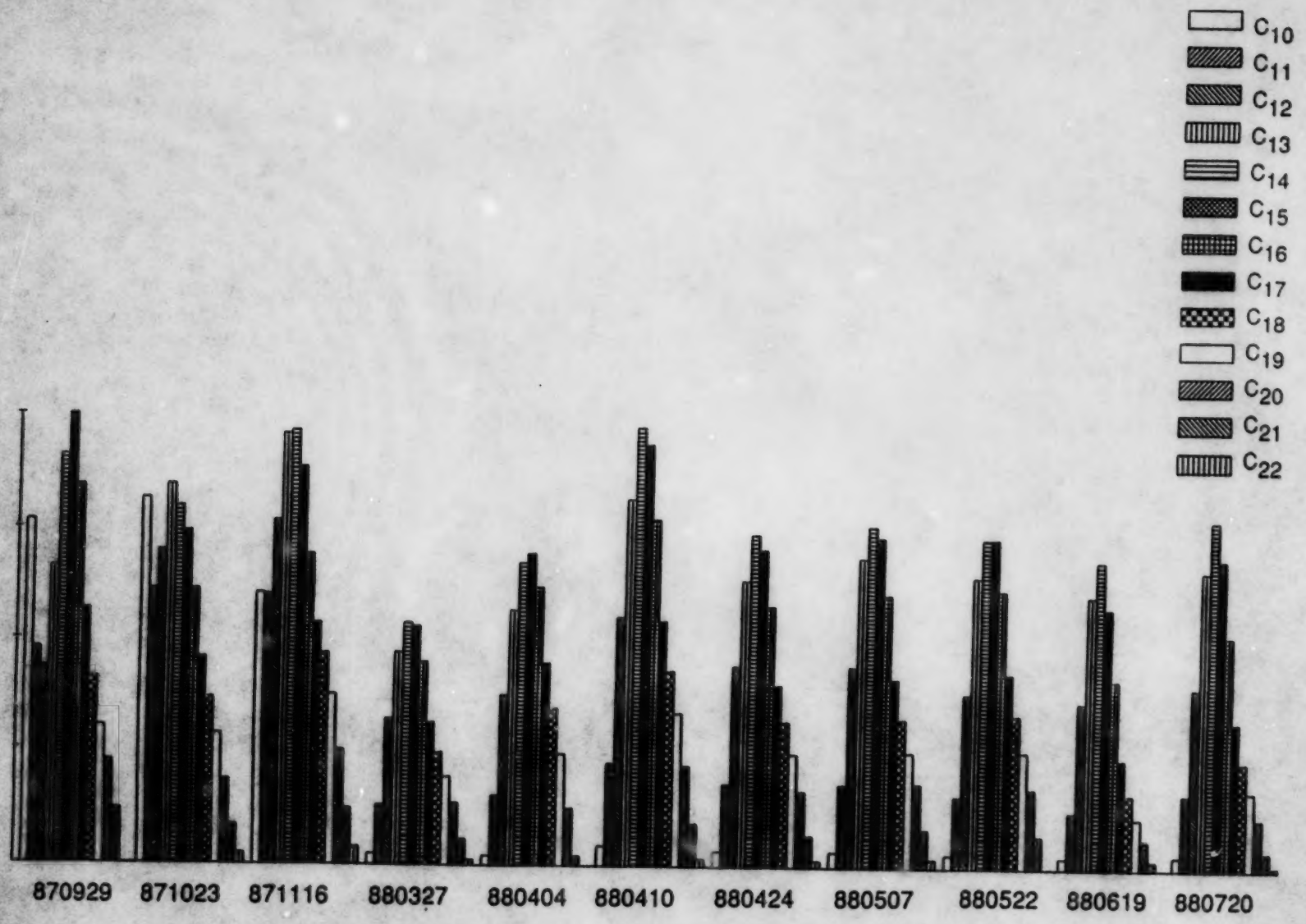


Figure 9. GC-MS analysis of the open chain saturate compound class from fraction 1 of the middle distillate cut as a function of time.

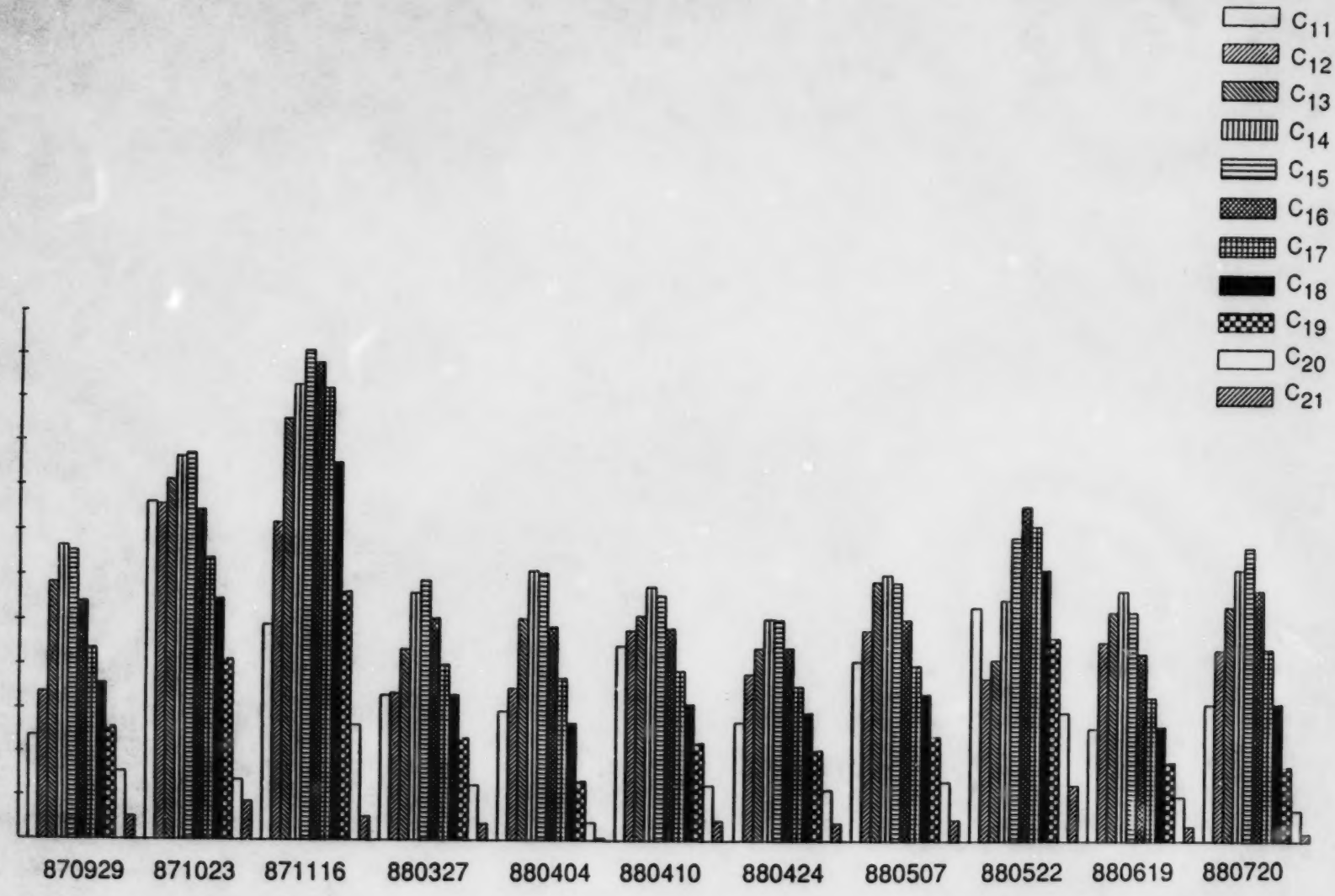


Figure 10. GC-MS analysis of the alkyl benzene compound class from fraction 2 of the middle distillate cut as a function of time.

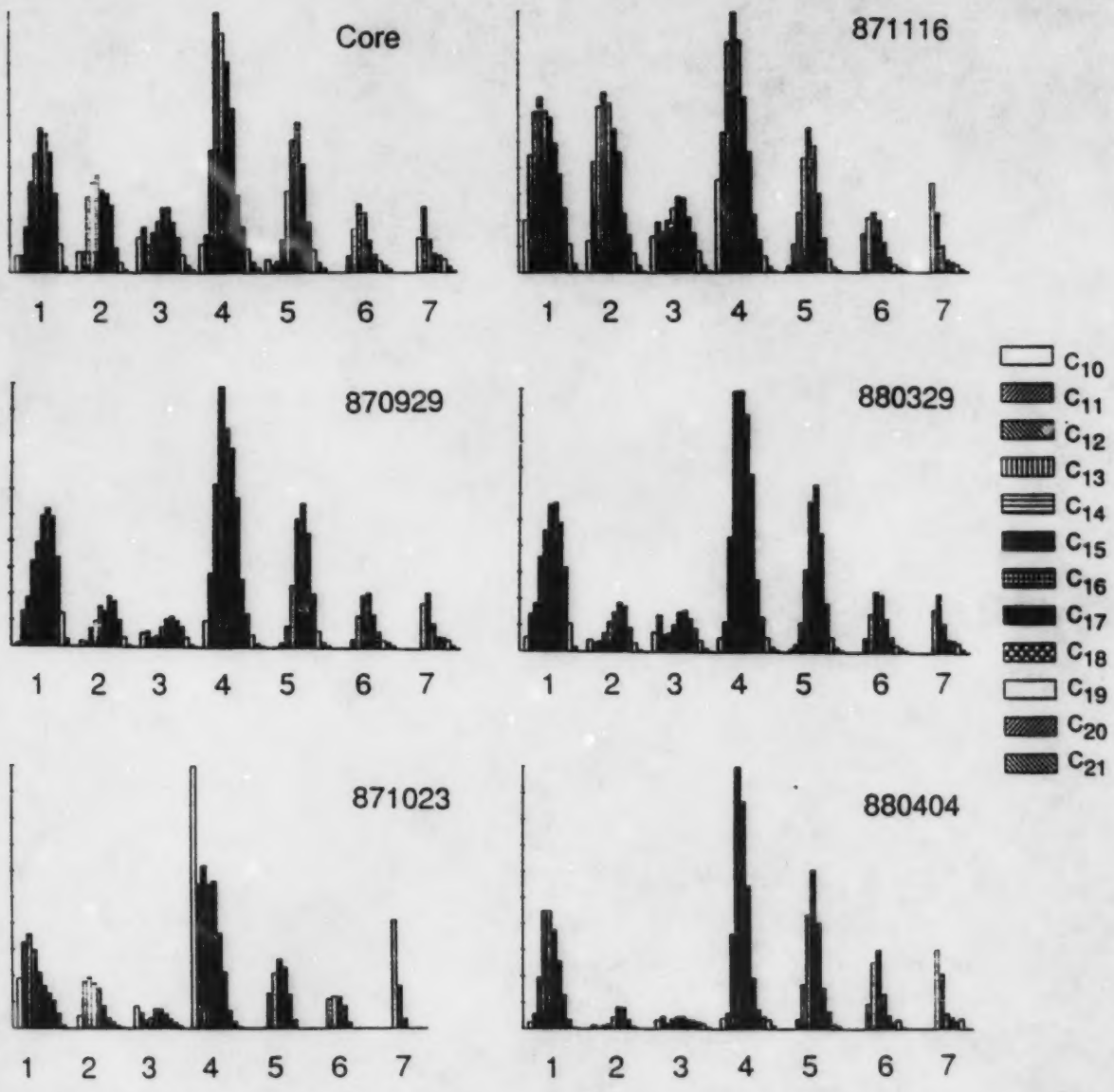


Figure 11. GC-MS analysis of the distribution of compound classes in fraction 3 of the middle distillate cuts.

1= alkyl benzenes
2= benzocycloparaffins
3= benzodicycloparaffins
4= naphthalenes
5= naphthocycloparaffins
6= fluorenes
7= phenanthrenes/anthracenes

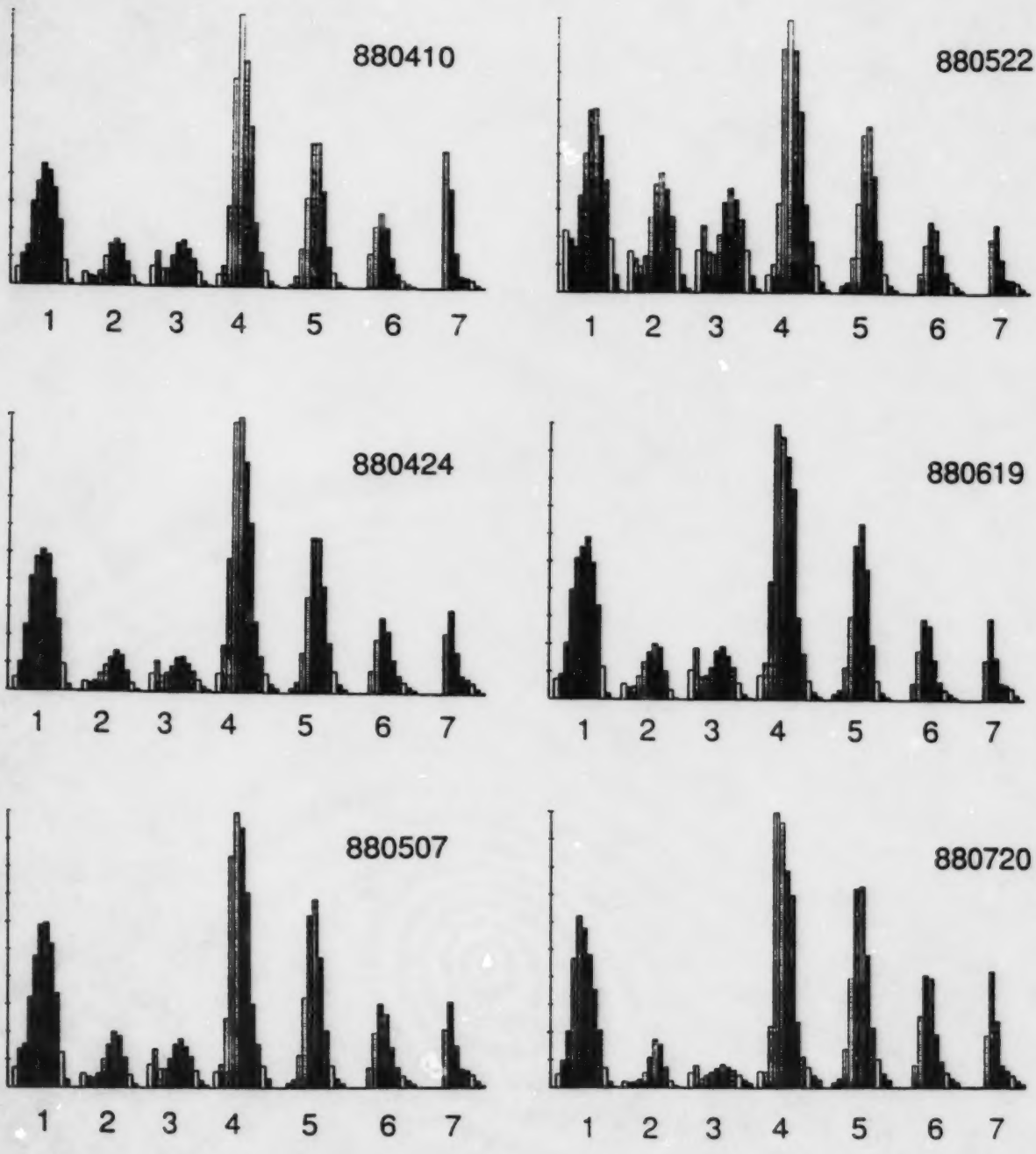


Figure 11. (cont.)

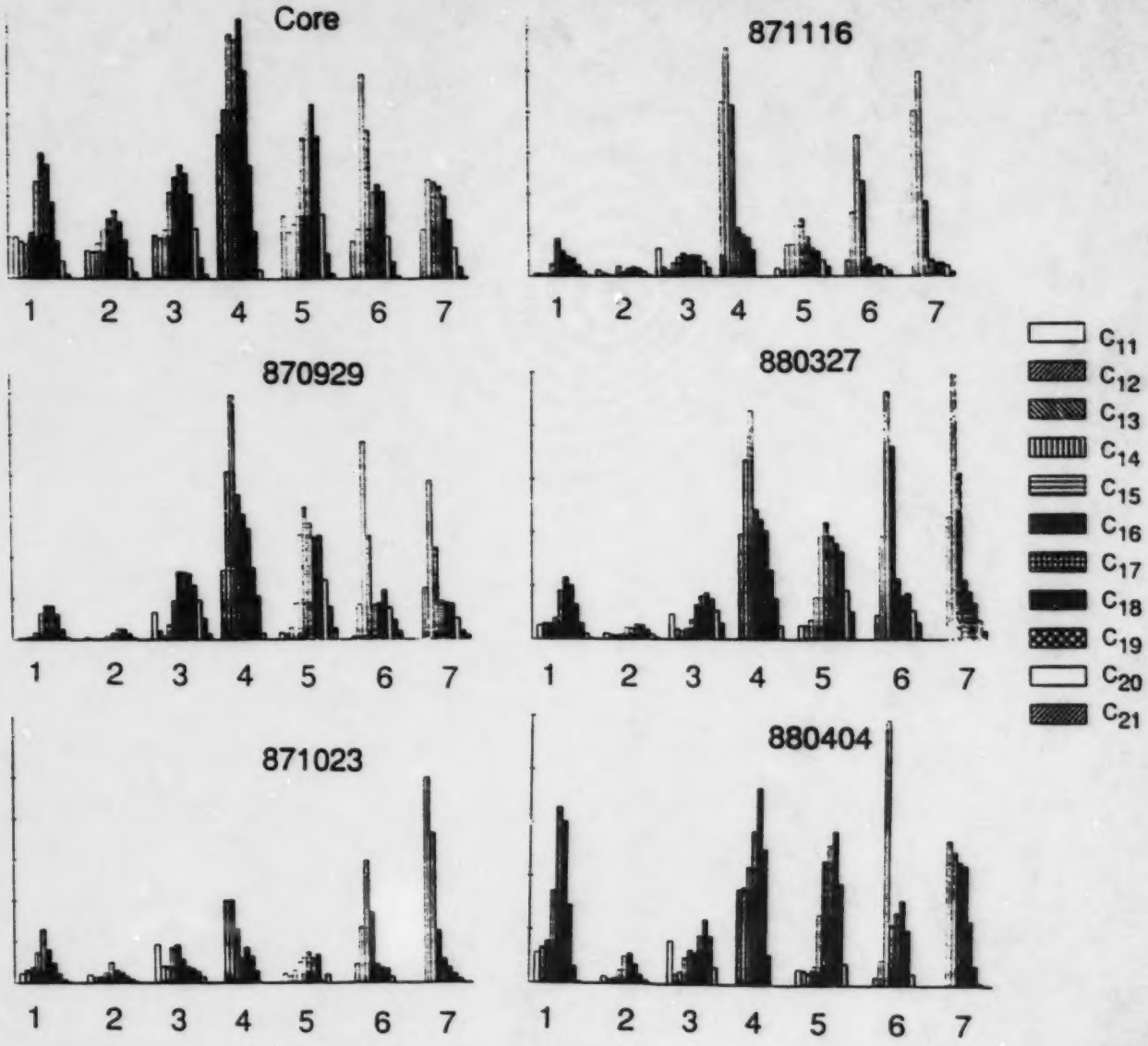


Figure 12. GC-MS analysis of the distribution of compound classes in fraction 4 of the middle distillate cuts (see text).

1= alkyl benzenes
2= benzocycloparaffins
3= benzodicycloparaffins
4= dibenzothiophenes
5= naphthocycloparaffins
6= fluorenes
7= phenanthrenes/ anthracenes

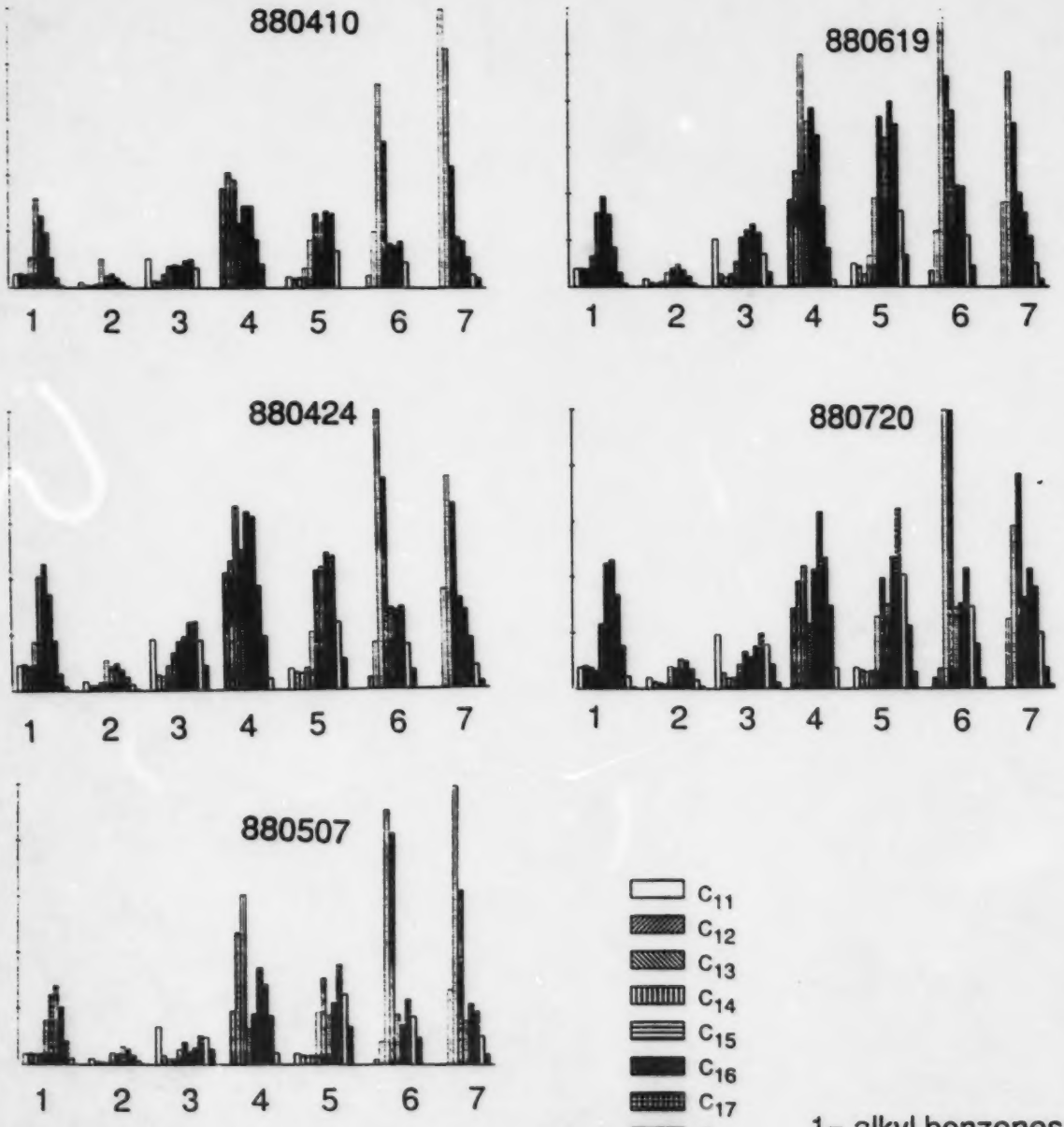


Figure 12. (cont.)

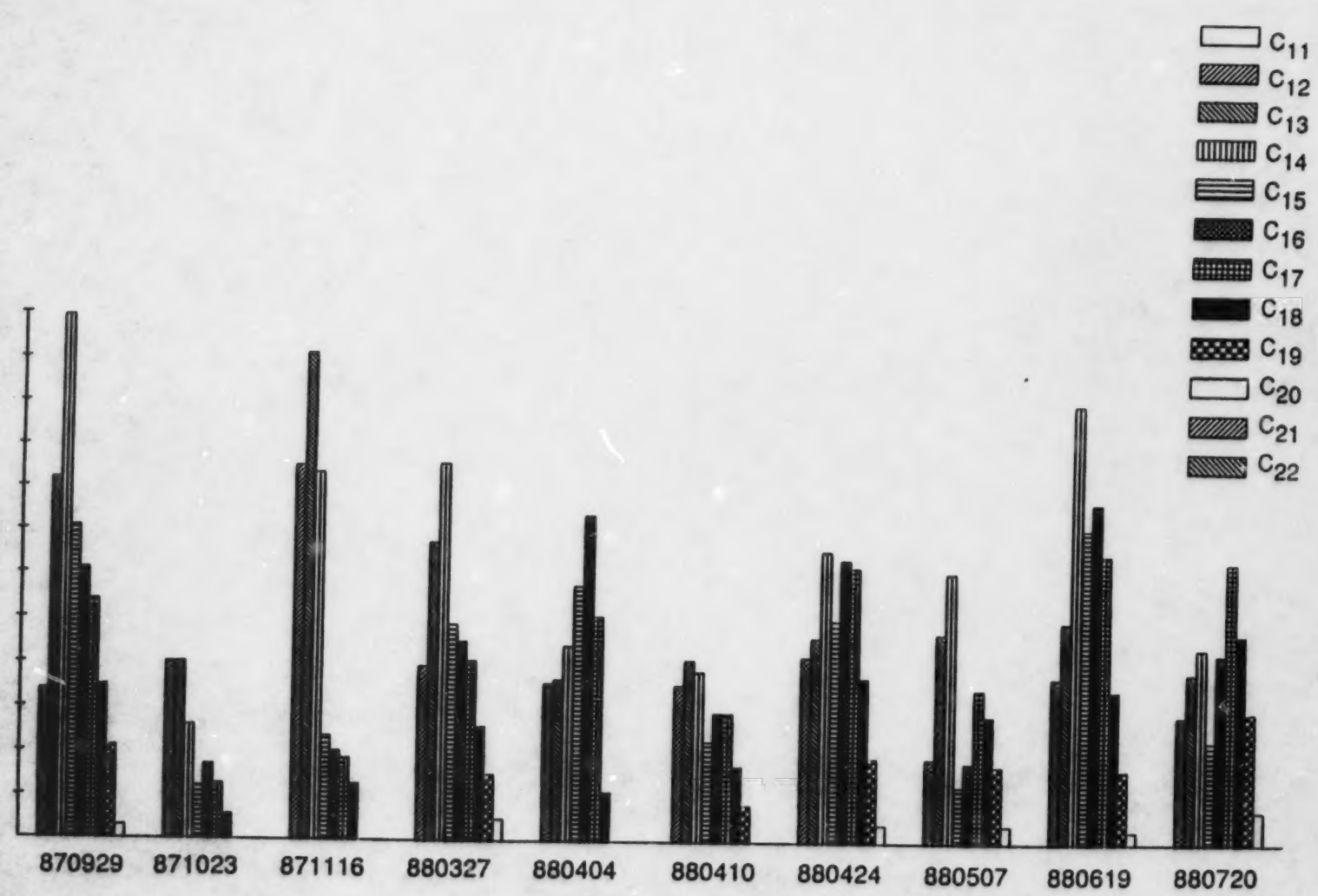


Figure 13. GC-MS analysis of the dibenzothiophene compound class from fraction 4 of the middle distillate cut as a function of time.

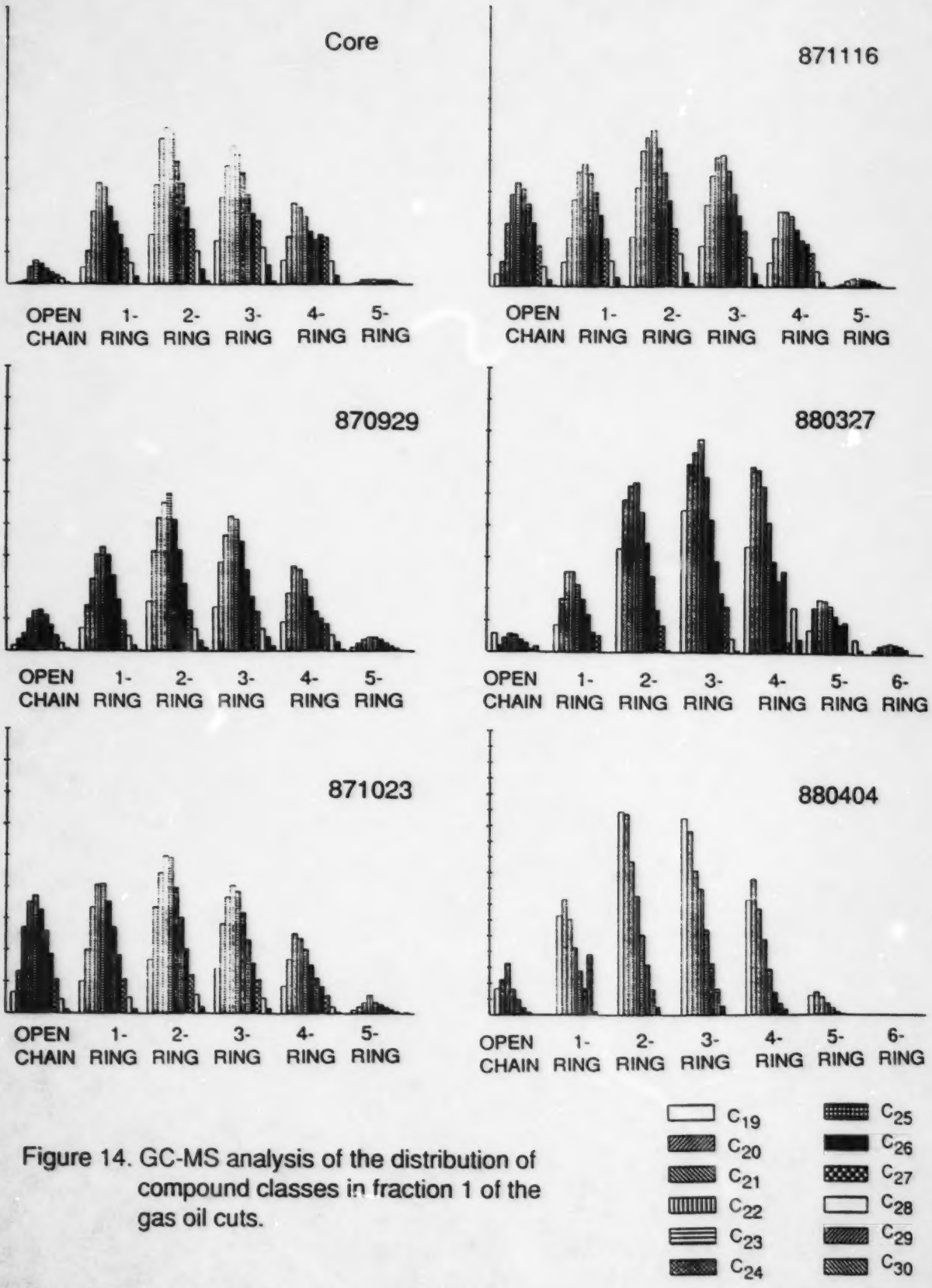


Figure 14. GC-MS analysis of the distribution of compound classes in fraction 1 of the gas oil cuts.

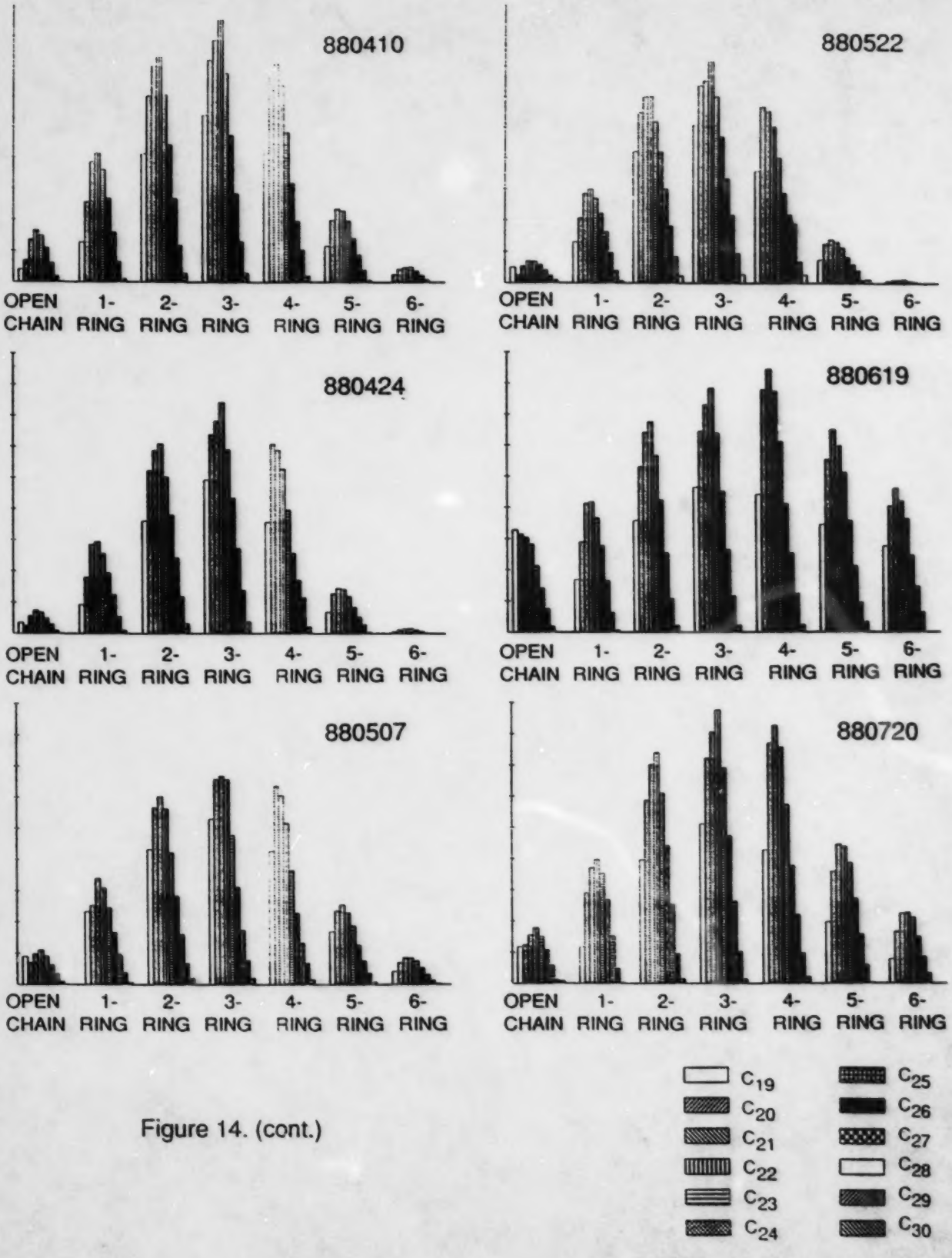


Figure 14. (cont.)

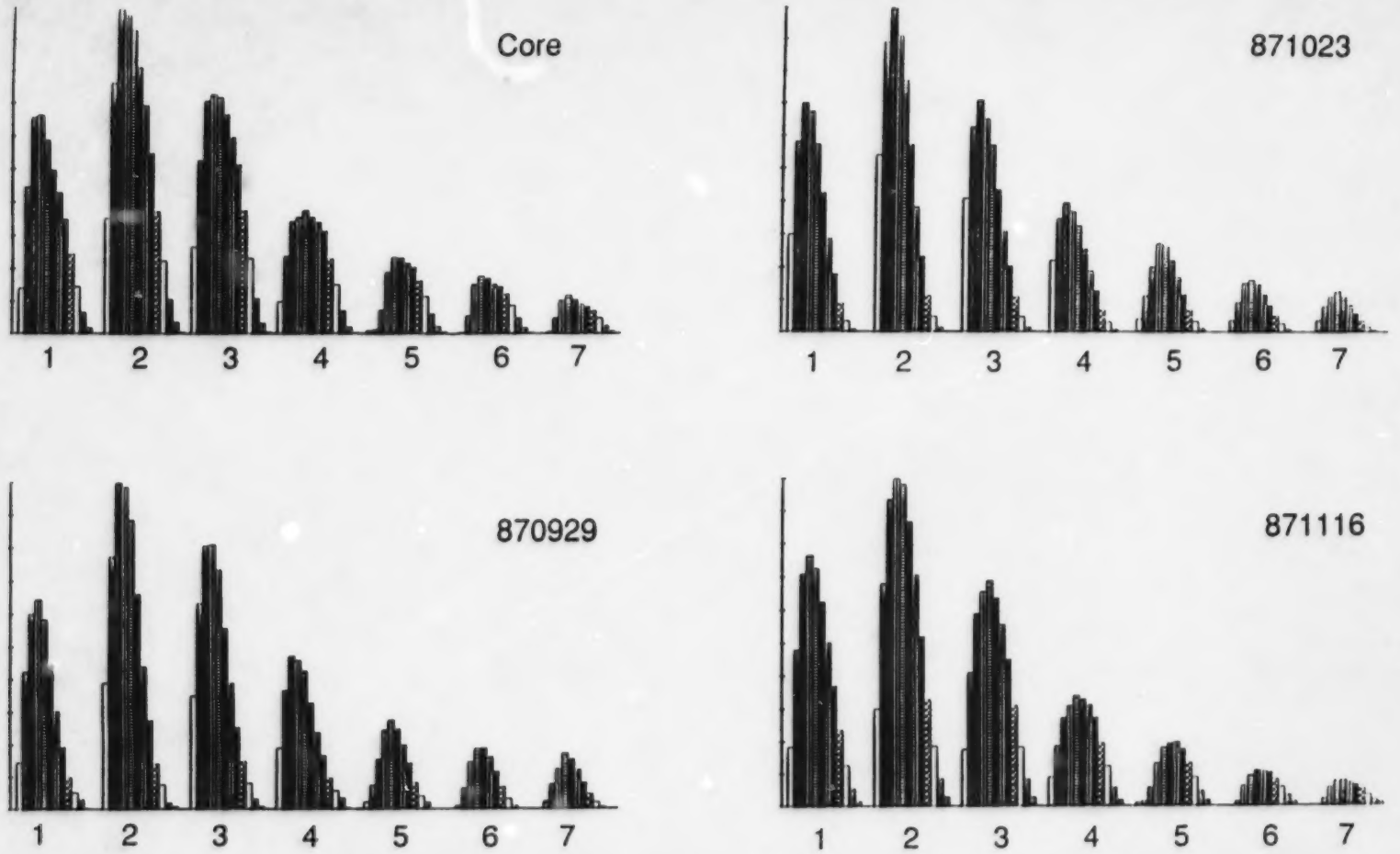


Figure 15. GC-MS analysis of the distribution of compound classes in fraction 2 of the gas oil cuts.

1= alkyl benzenes
2= benzocycloparaffins
3= benzodicycloparaffins
4= naphthalenes
5= naphthocycloparaffins
6= fluorenes
7= phenanthrenes/ anthracenes

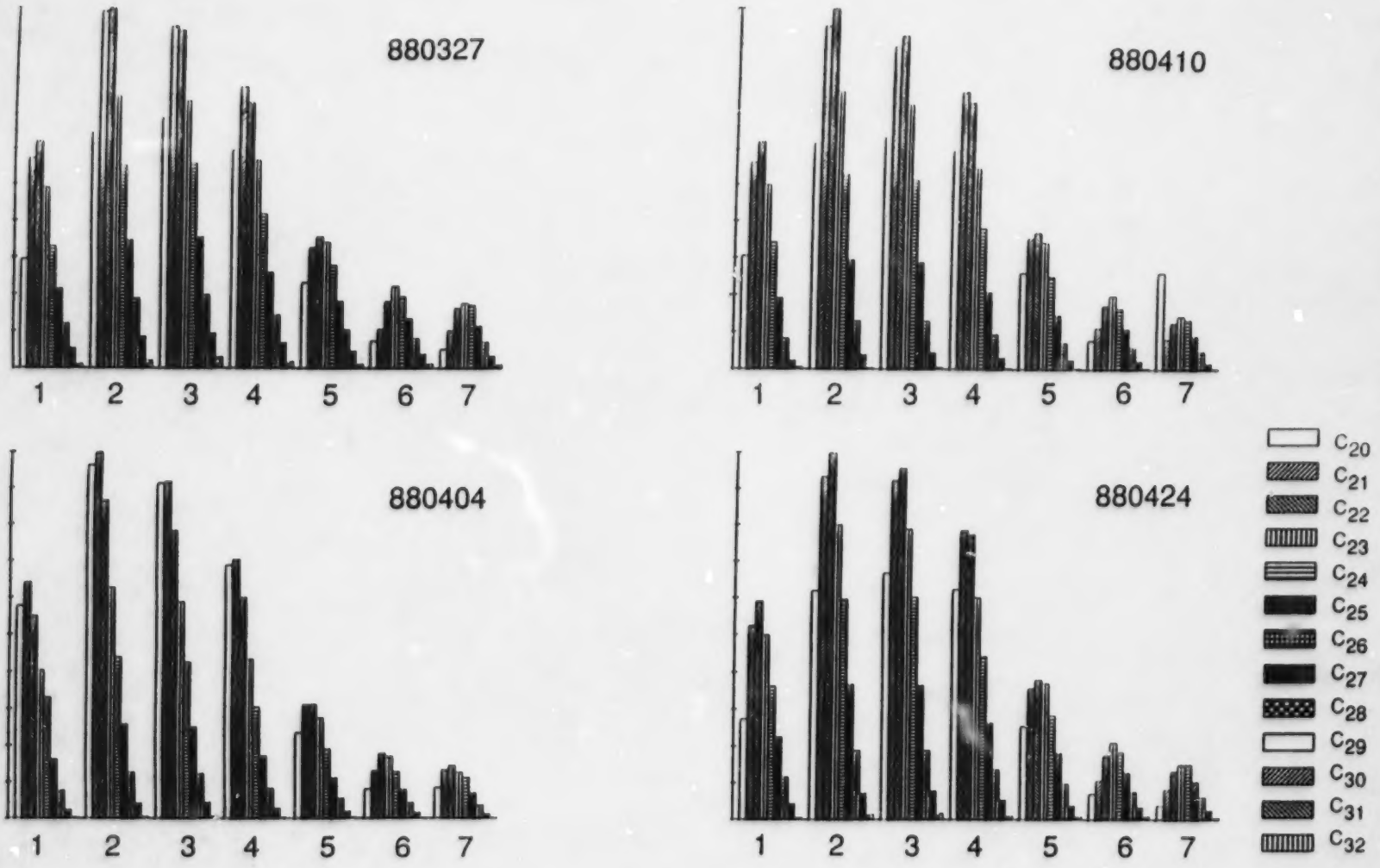


Figure 15. (cont.)

1= alkyl benzenes
2= benzocycloparaffins
3= benzodicycloparaffins
4= naphthalenes
5= naphthocycloparaffins
6= fluorenes
7= phenanthrenes/ anthracenes

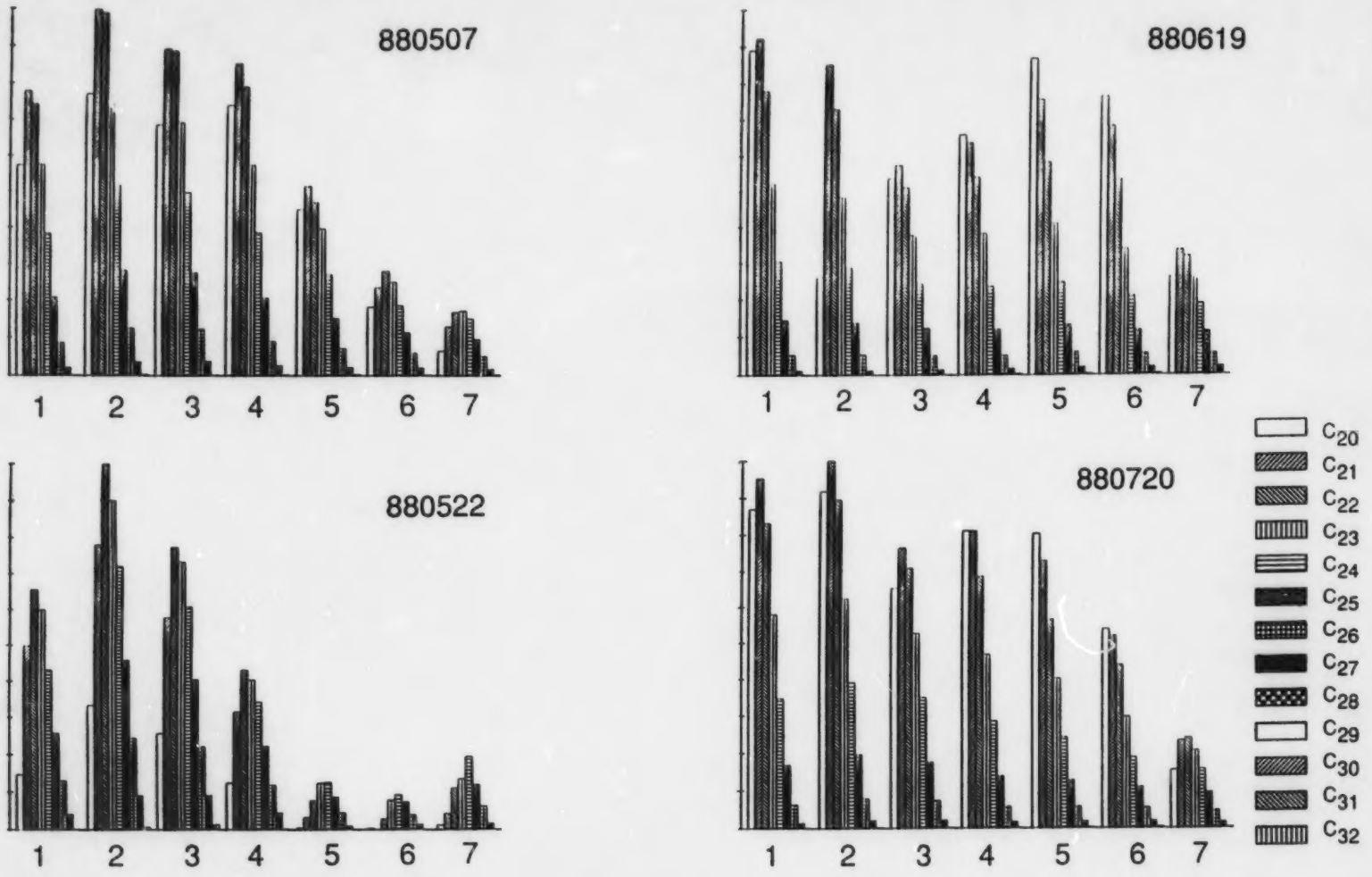


Figure 15. (cont.)

1= alkyl benzenes
2= benzocycloparaffins
3= benzodicycloparaffins
4= naphthalenes
5= naphthocycloparaffins
6= fluorenes
7= phenanthrenes/ anthracenes

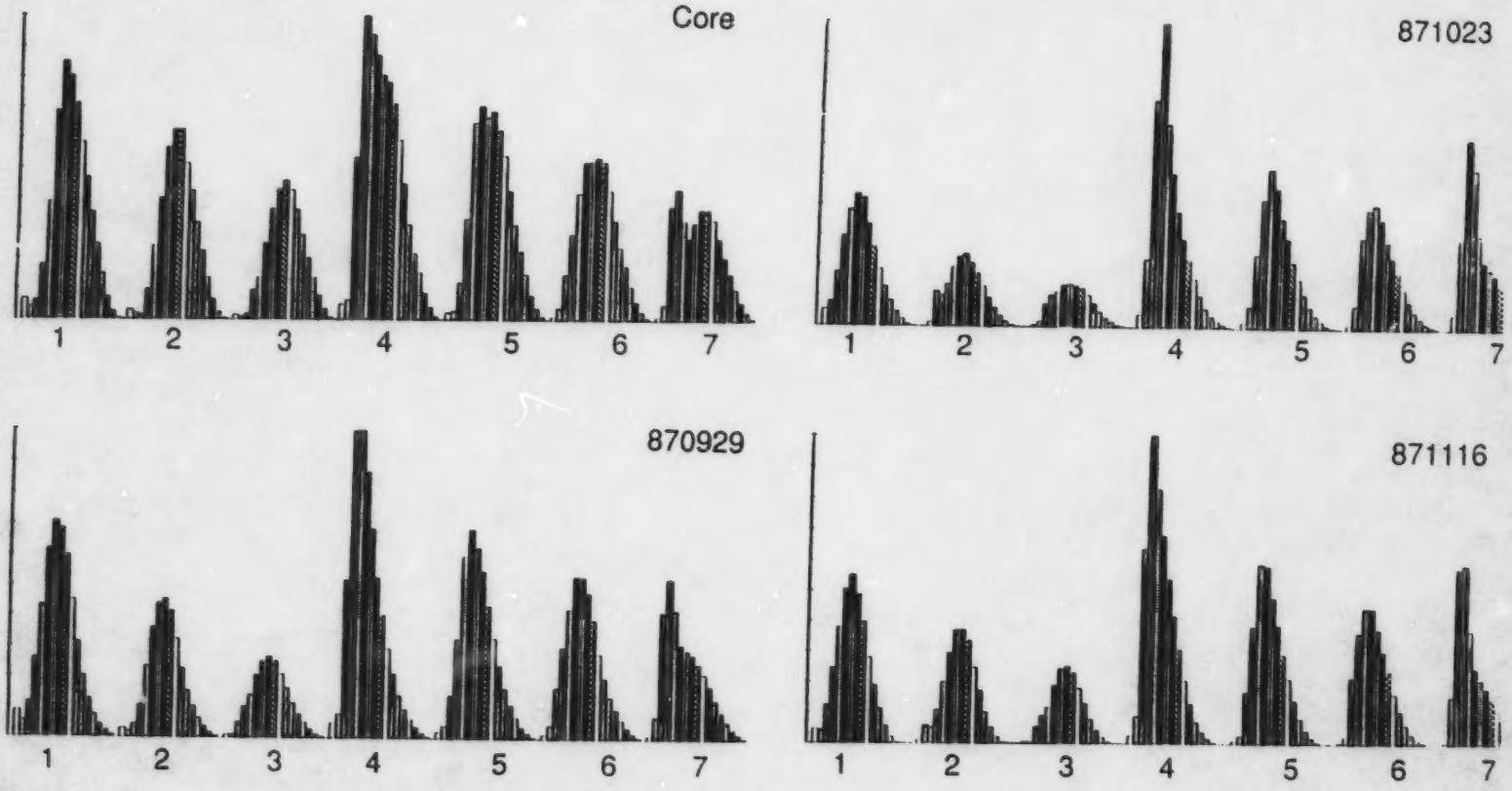


Figure 16. GC-MS analysis of the distribution of compound classes in fraction 3 of the gas oil cuts.

1= alkyl benzenes
2= benzocycloparaffins
3= benzodicycloparaffins
4= naphthalenes
5= naphthocycloparaffins
6= fluorenes
7= phenanthrenes/ anthracenes

C_{14} C_{15} C_{16} C_{17} C_{18} C_{19} C_{20} C_{21} C_{22} C_{23} C_{24} C_{25} C_{26} C_{27} C_{28} C_{29} C_{30}

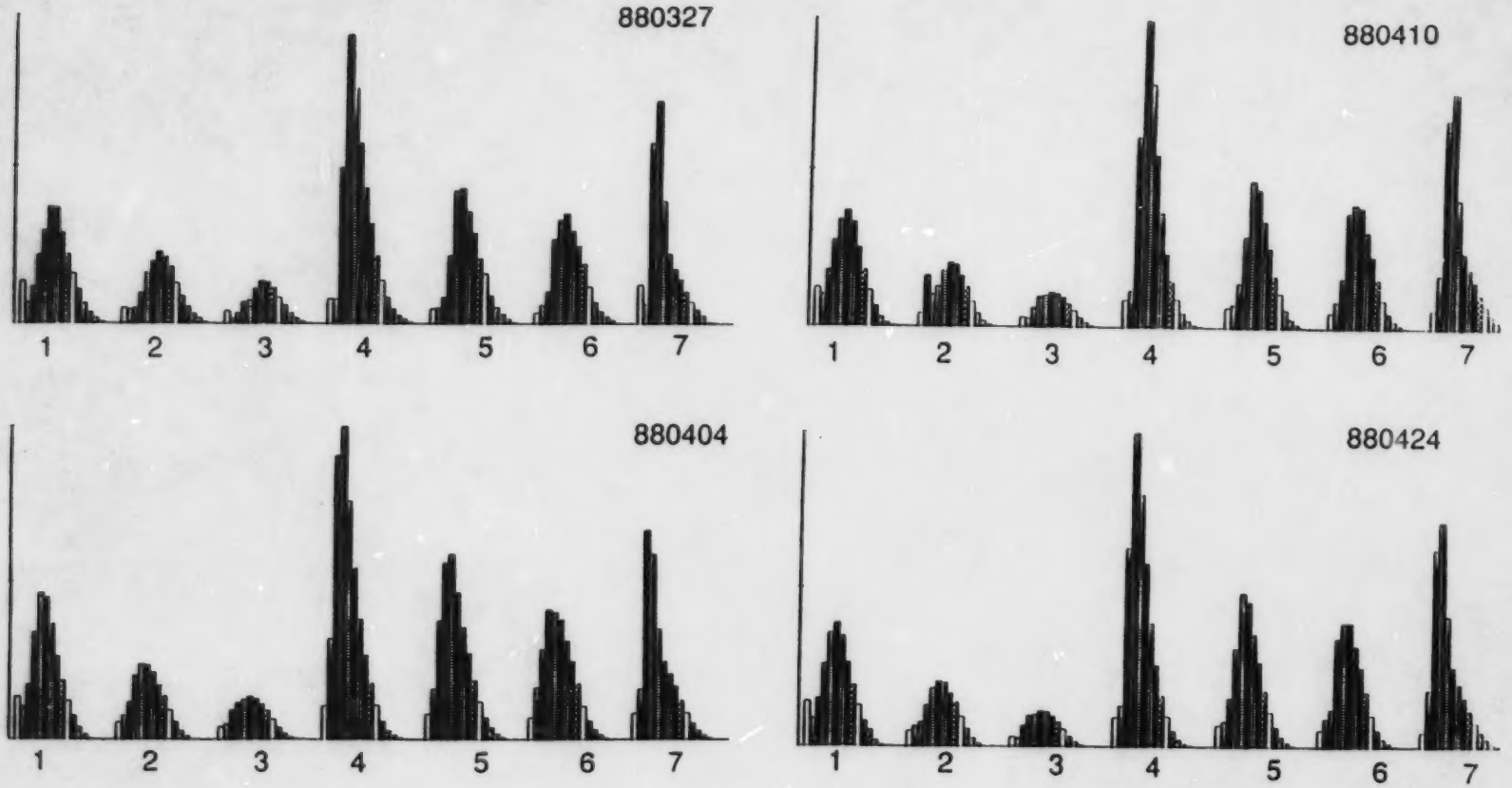


Figure 16. (cont.)

1= alkyl benzenes
2= benzocycloparaffins
3= benzodicycloparaffins
4= naphthalenes
5= naphthocycloparaffins
6= fluorenes
7= phenanthrenes/ anthracenes

C_{14} C_{15} C_{16} C_{17} C_{18} C_{19} C_{20} C_{21} C_{22} C_{23} C_{24} C_{25} C_{26} C_{27} C_{28} C_{29} C_{30}

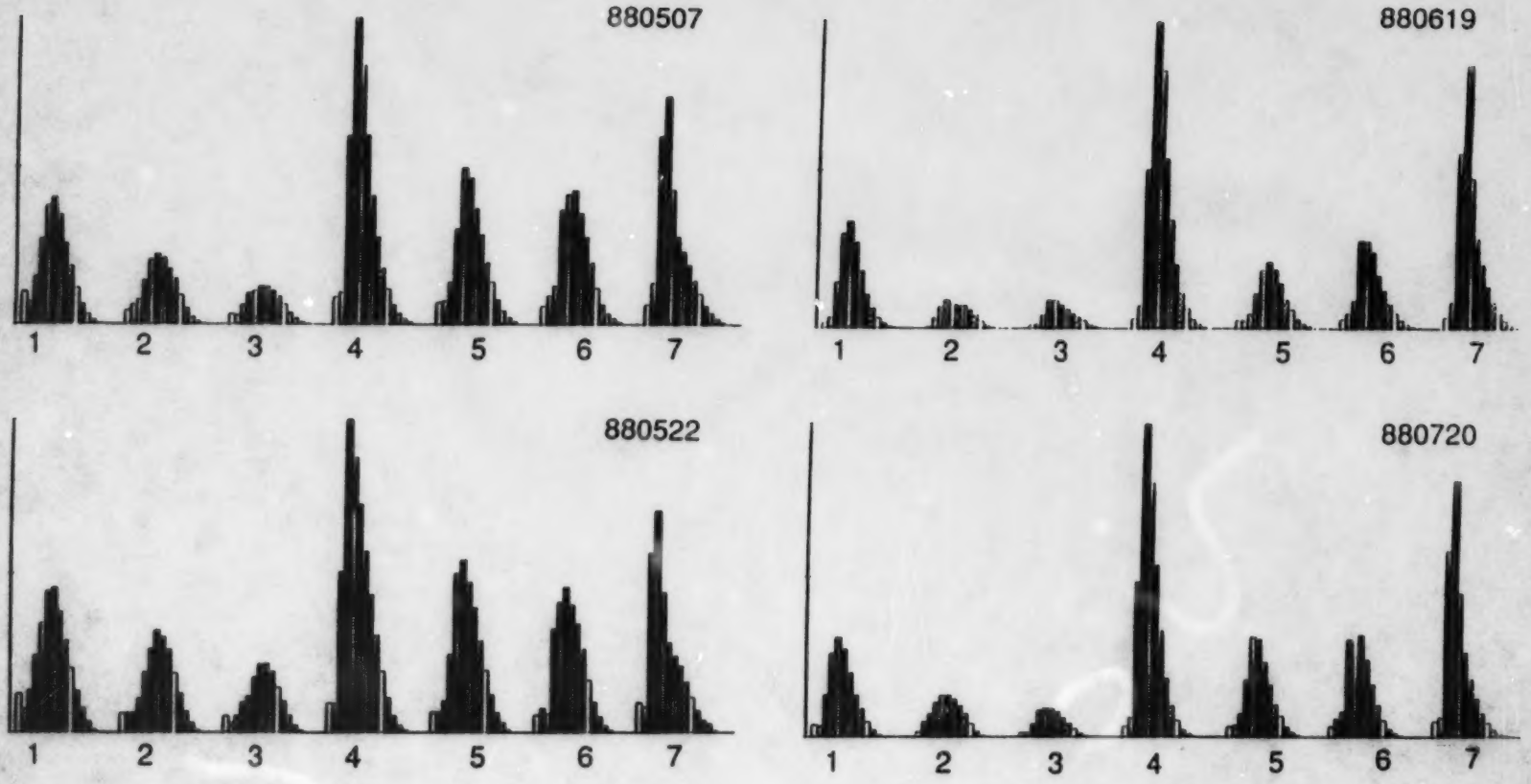


Figure 16. (cont.)

1= alkyl benzenes
2= benzocycloparaffins
3= benzodicycloparaffins
4= naphthalenes
5= naphthocycloparaffins
6= fluorenes
7= phenanthrenes/ anthracenes

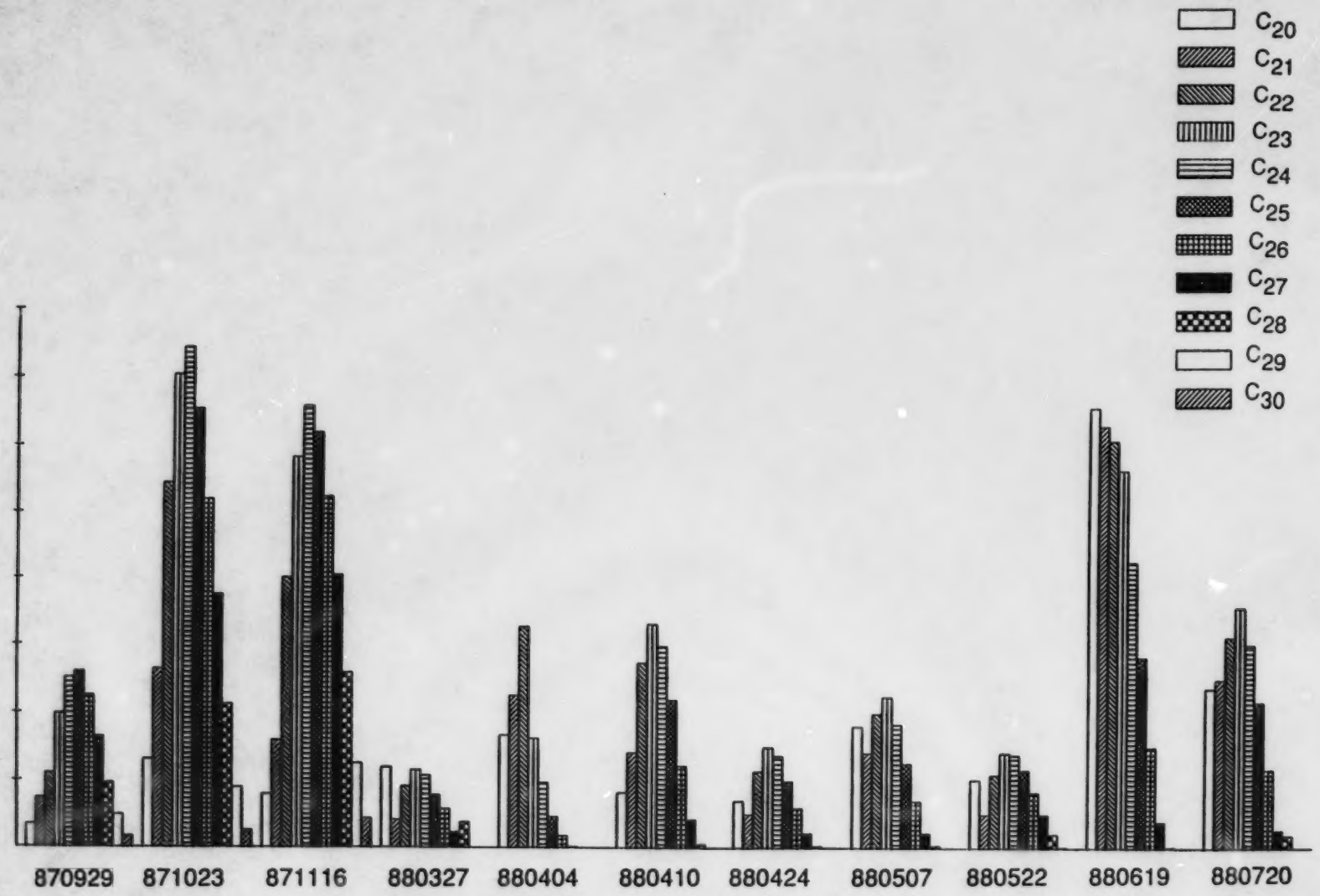


Figure 17. GC-MS analysis of the open chain saturate compound class from fraction 1 of the gas oil cut as a function of time.

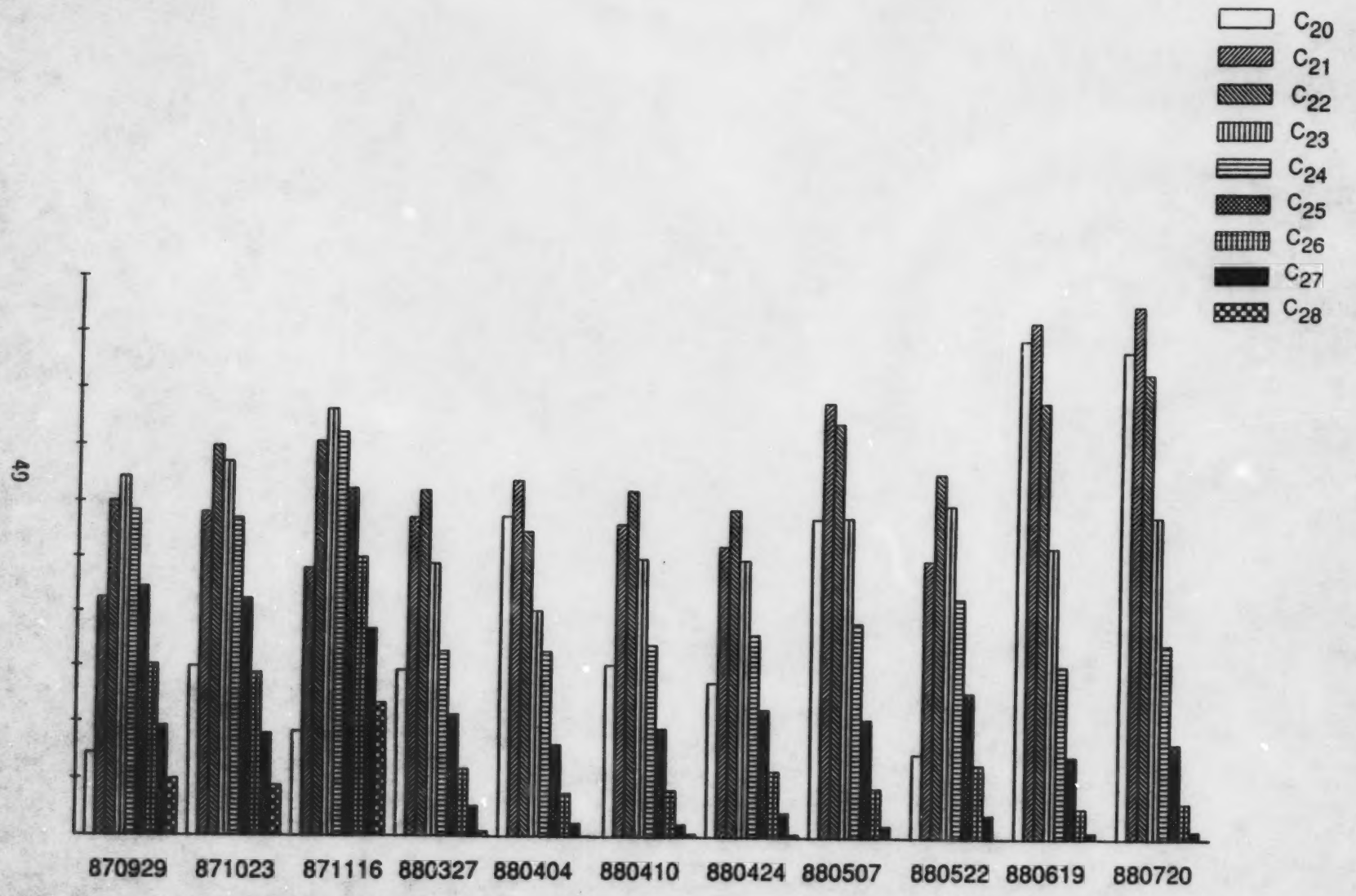


Figure 18. GC-MS analysis of the alkyl benzene compound class from fraction 2 of the gas oil cut as a function of time.

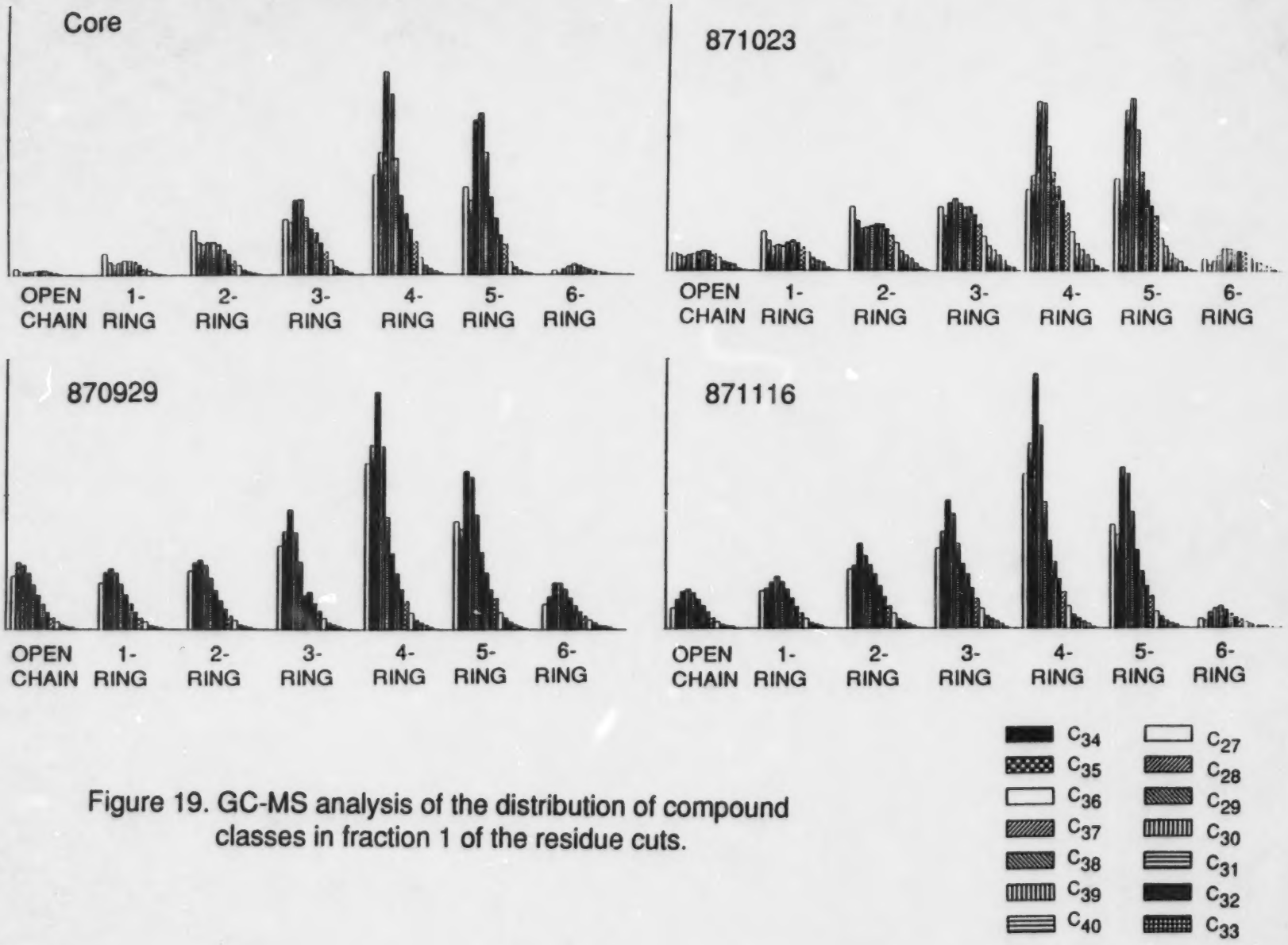


Figure 19. GC-MS analysis of the distribution of compound classes in fraction 1 of the residue cuts.

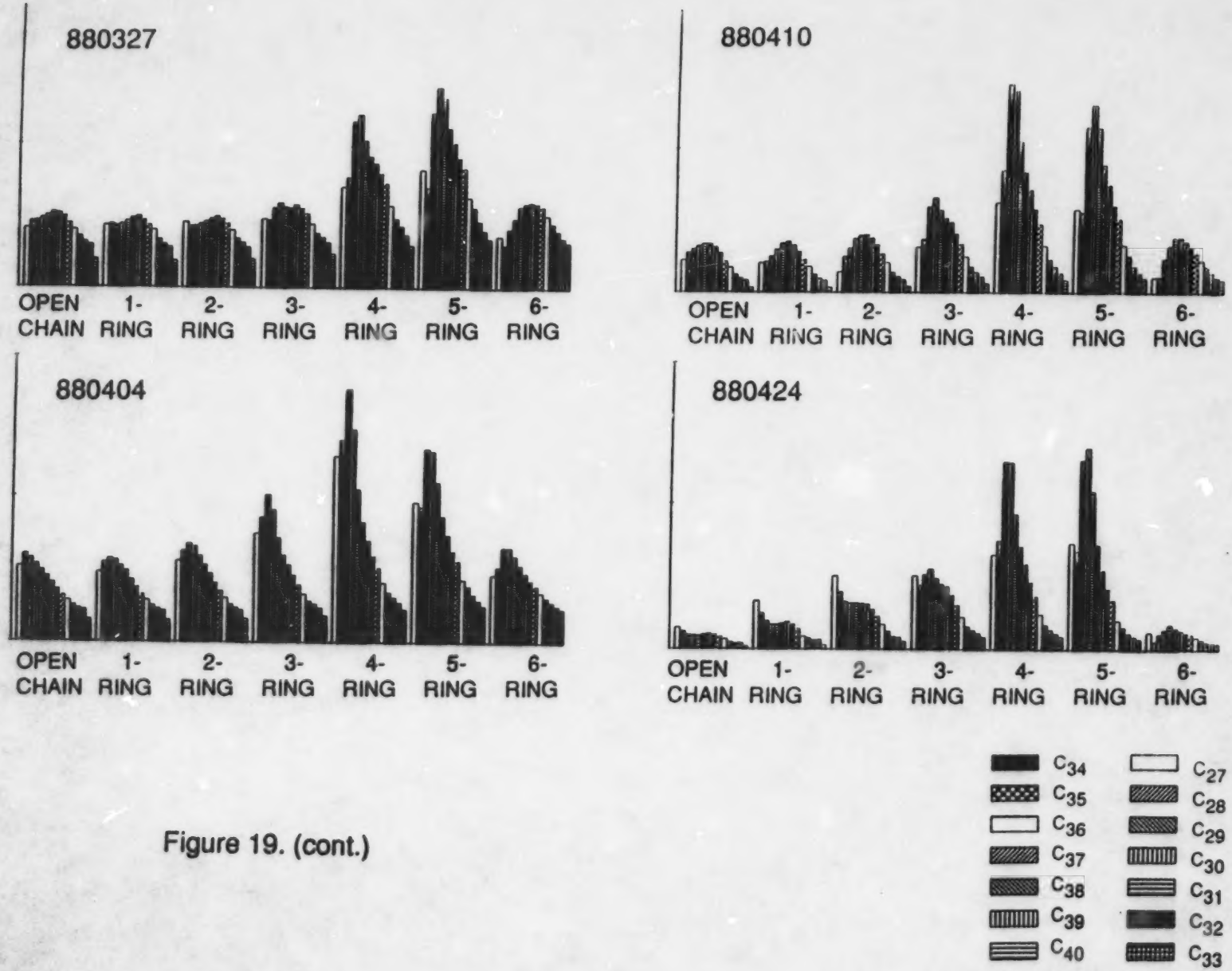


Figure 19. (cont.)

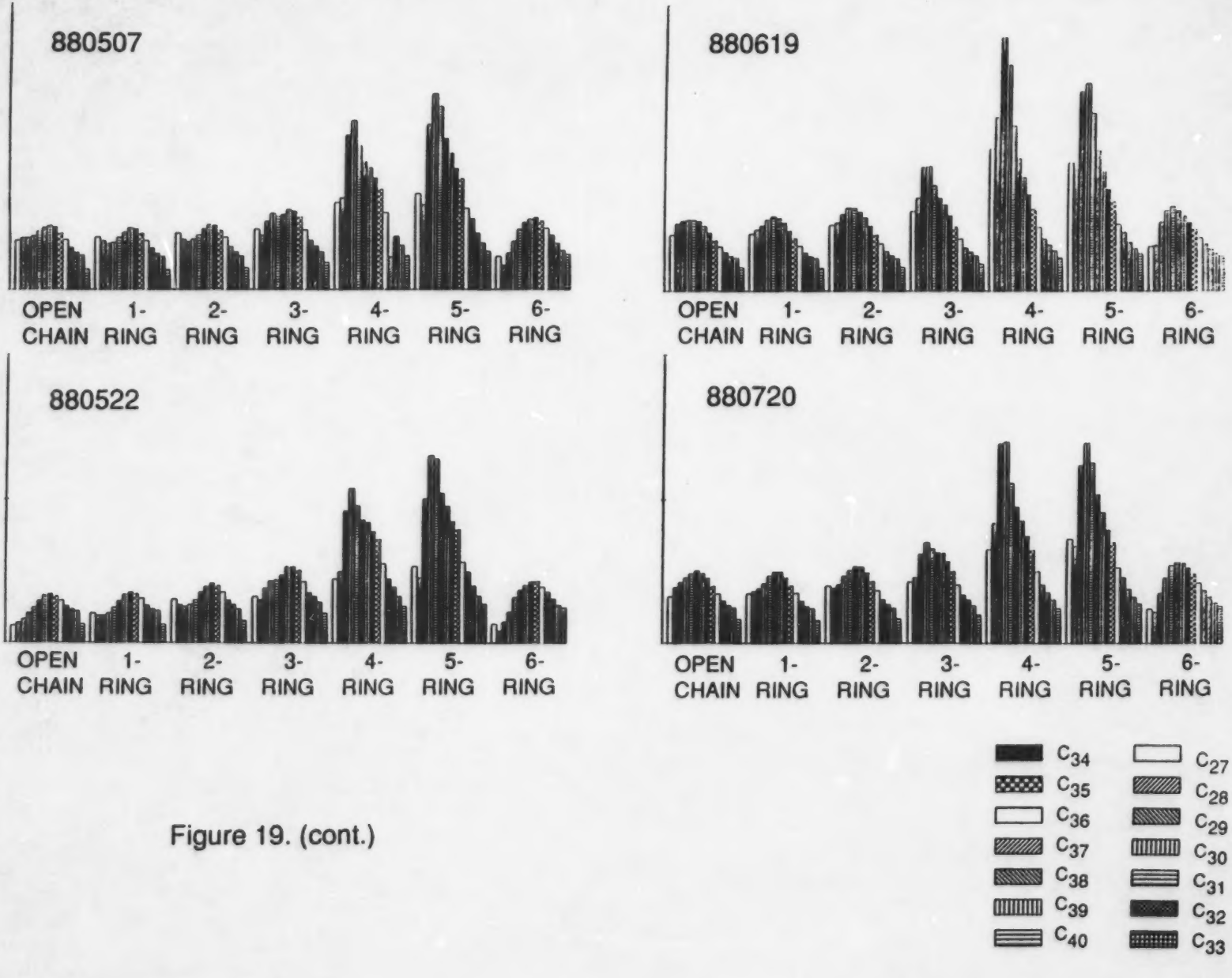


Figure 19. (cont.)

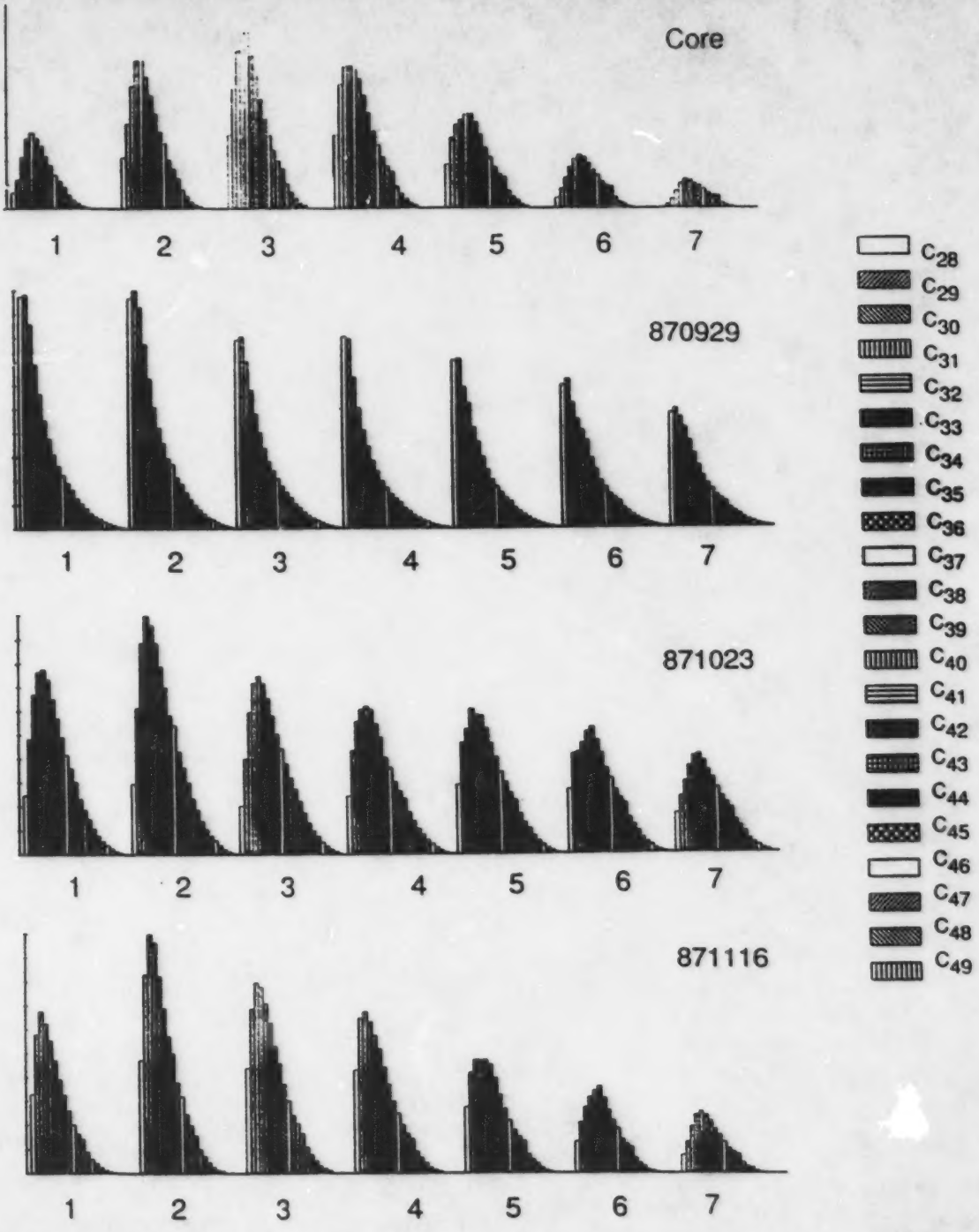


Figure 20. GC-MS analysis of the distribution of compound classes in fraction 2 of the residue cuts.

1= alkyl benzenes
2= benzocycloparaffins
3= benzodicycloparaffins
4= naphthalenes
5= naphthocycloparaffins
6= fluorenes
7= phenanthrenes/anthracenes

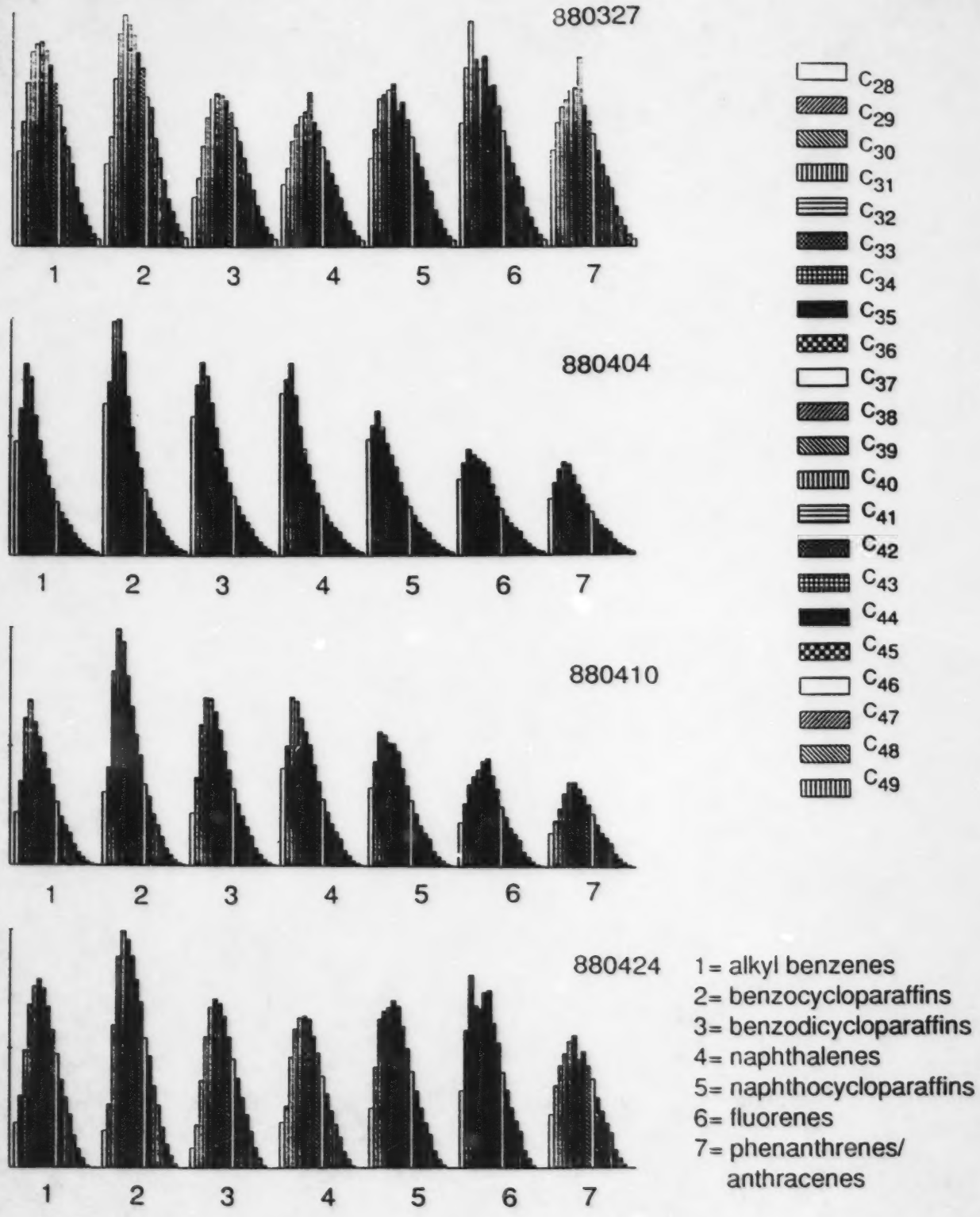


Figure 20. (cont.)

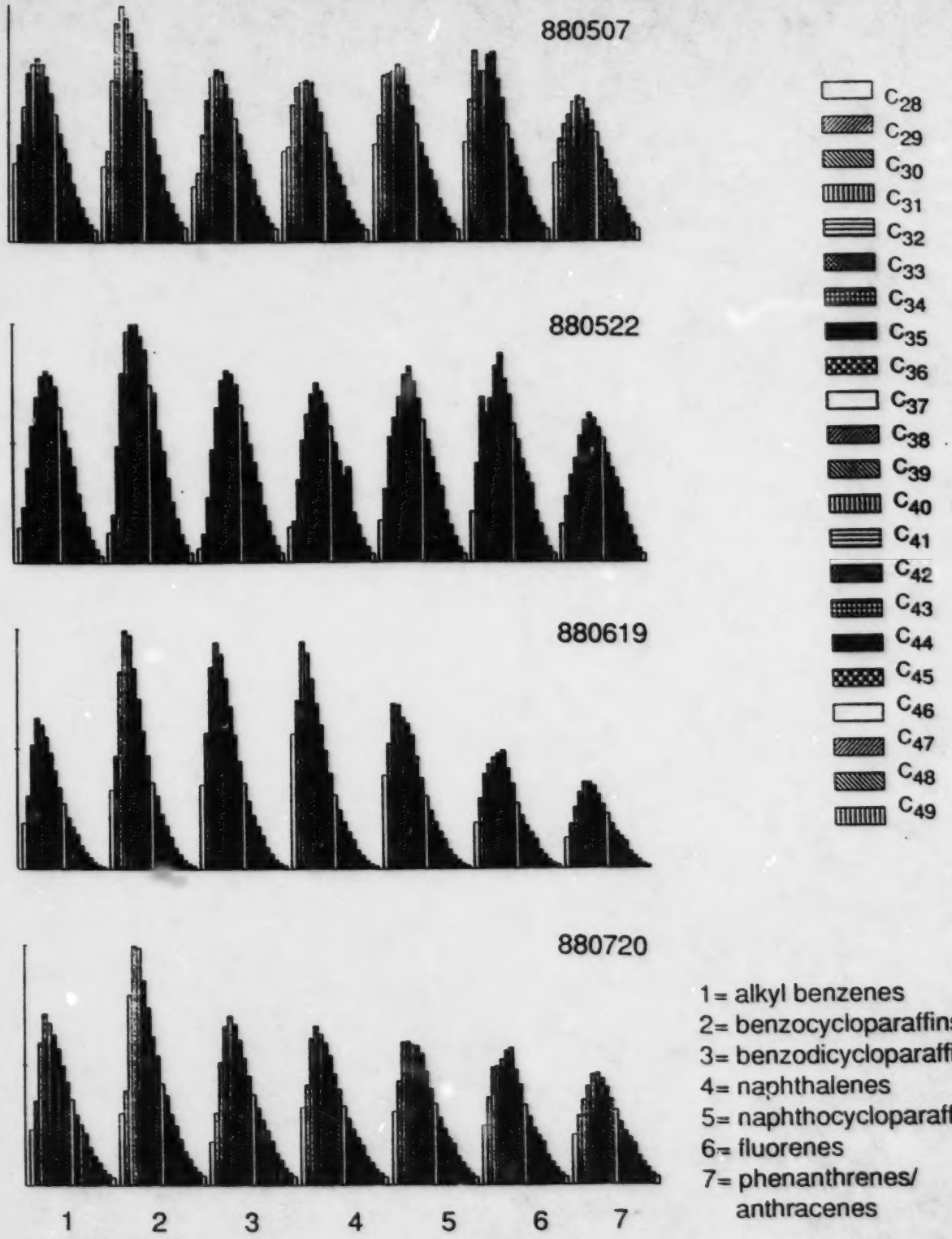


Figure 20. (cont.)

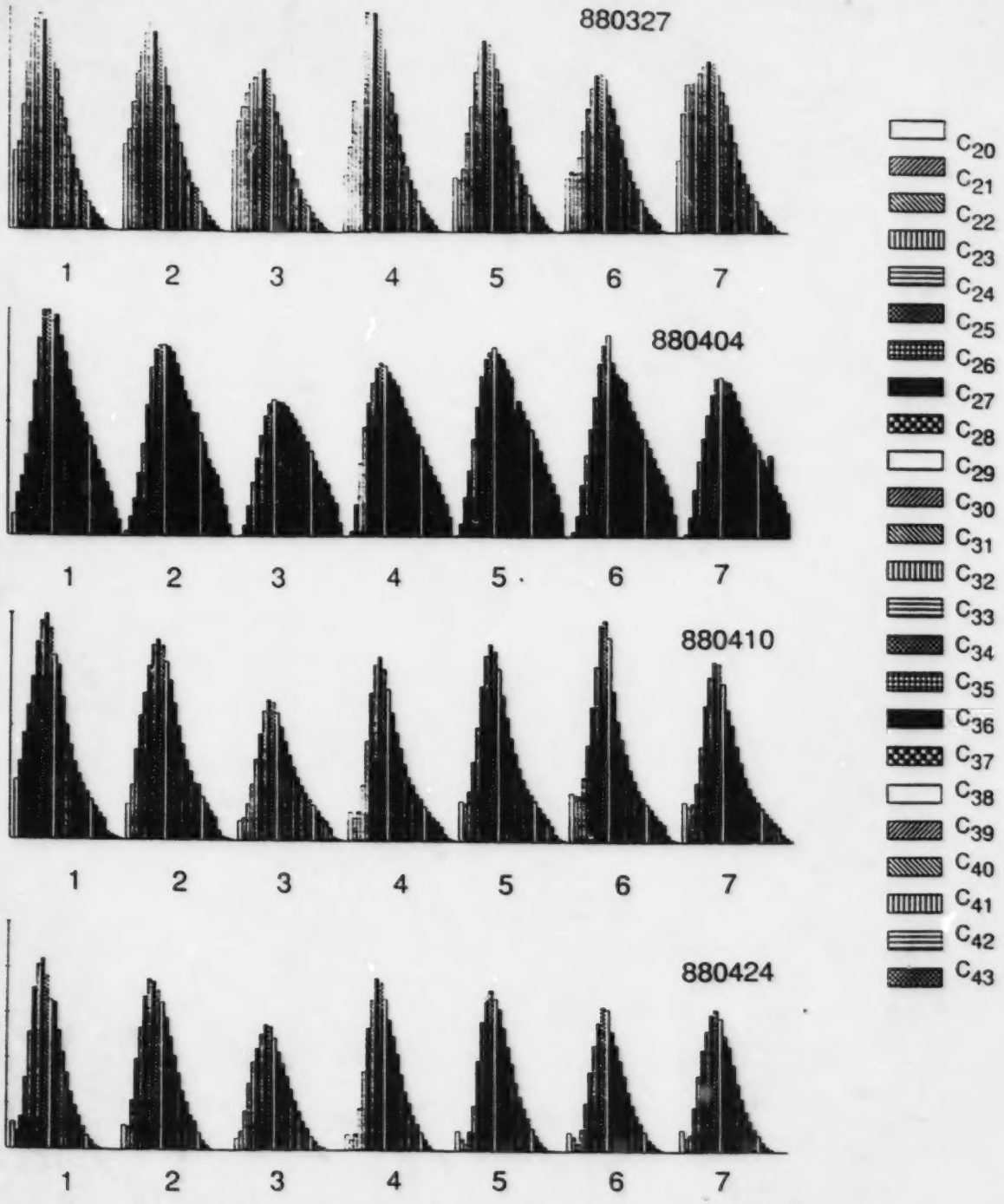


Figure 21. GC-MS analysis of the distribution of compound classes in fraction 3 of the residue cuts (phase 2 samples).

1= alkyl benzenes
2= benzocycloparaffins
3= benzodicycloparaffins
4= naphthalenes
5= naphthocycloparaffins
6= fluorenes
7= phenanthrenes/anthracenes

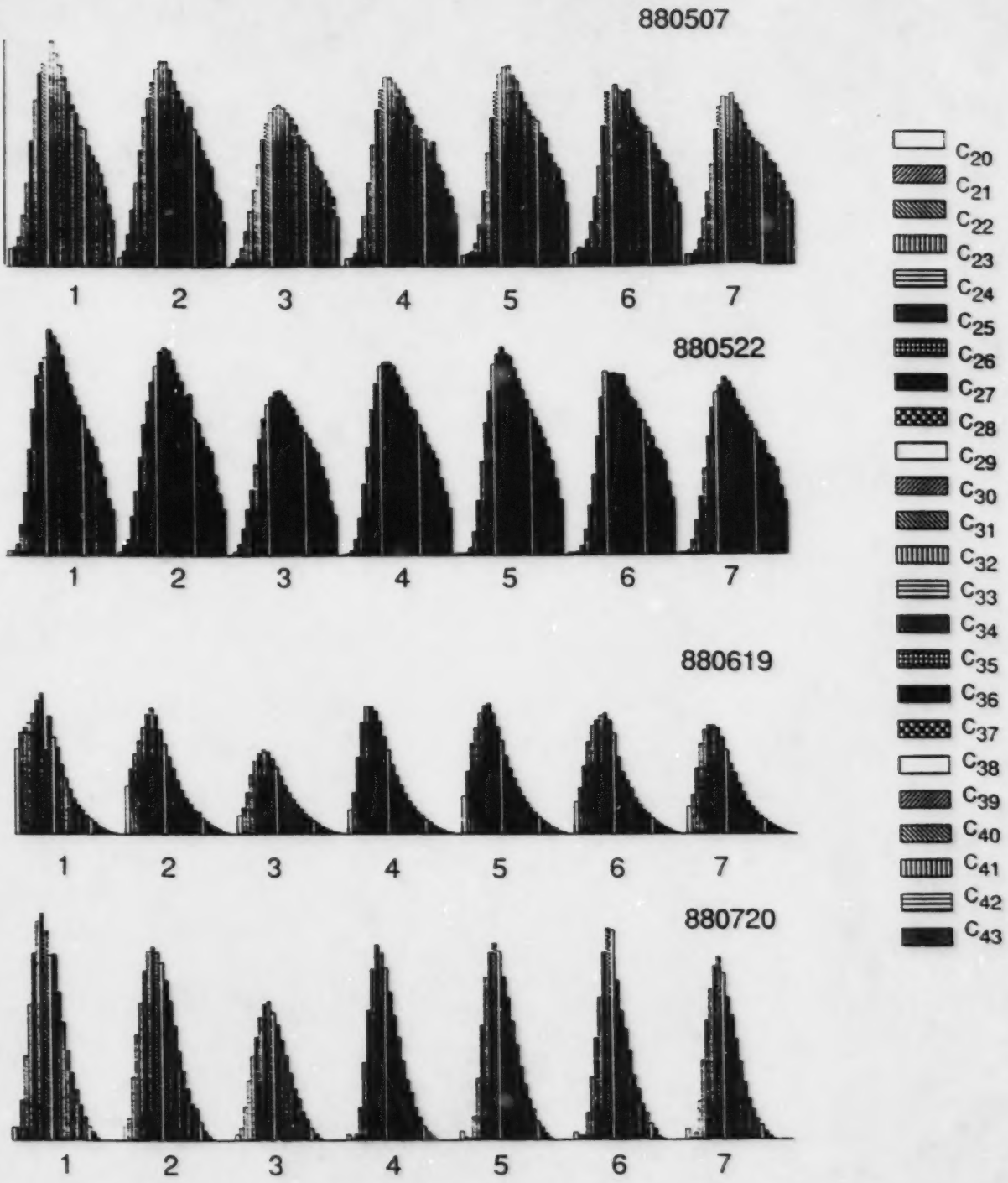


Figure 21. (cont.)

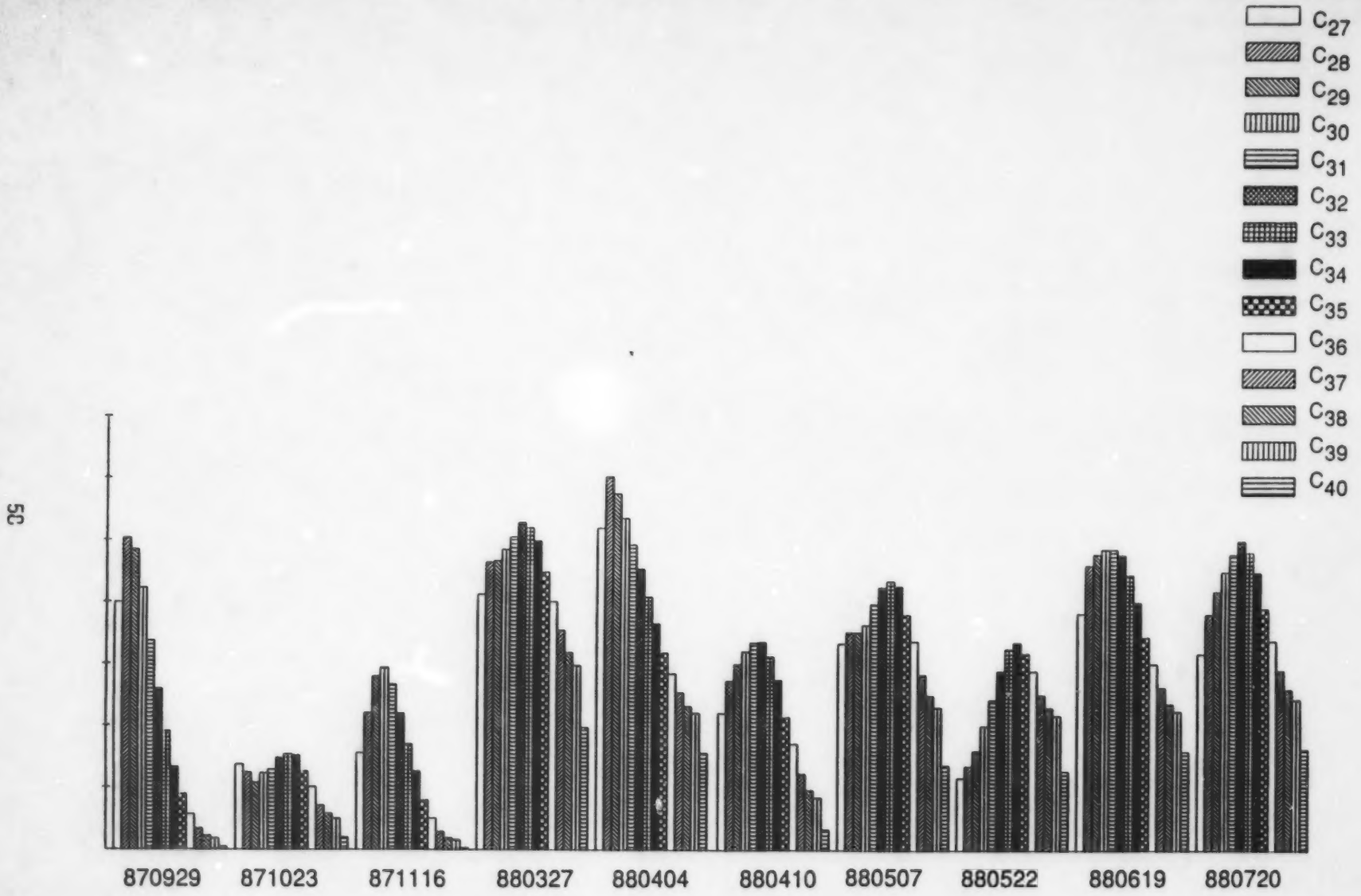


Figure 22. GC-MS analysis of the open chain saturate compound class from fraction 1 of the residue cut as a function of time.

Table 1: Analytical Data for Whole Oil Samples

Sample	Density (g/mL, 25°C)	Viscosity T(°C)	Viscosity (cP)	Water[a] (Wt%)
Core	0.9894	25	49,400	
		30	26,500[b]	
		61	1,300	
		80	372	
870929	0.9888	25	12,700	0.07 (vol%)
		30	7,350[b]	
		61	557	
		80	175	
871023	0.9795	25	1,765	0.08 (vol%)
		30	1,150[b]	
		61	148	
		80	59	
871116	0.9688	25	820	0.05 (vol%)
		30	568[b]	
		61	93	
		80	39	
880327	0.9924	30	9,450	0.17
		80	208	
880404	0.9835	30	5,630	0.10
		80	155	
880410	0.9810	30	2,180	0.08
		80	88	
880424	0.9858	30	5,140	0.23
		80	143	
880507	0.9879	30	6,450	0.14
		80	170	
880522	0.9828	30	4,000	0.13
		80	132	
880619	0.9906	30	6,720	0.15
		80	170	
880720	0.9898	30	7,460	0.15
		80	179	

a: after ultracentrifugation
b: interpolated from viscosities at 25, 61, and 80 °C

Table 2: Distillation Data (Vol%) for Whole Oil Samples

SAMPLE	NAPHTHA (115-204°C)	MIDDLE DISTILLATE (204-345°C)	GAS OIL (345-442°C)	RESIDUE (>442°C)
Core	0.8*	17.7	16.5	65.0
870929	2.59	19.56	17.66	60.19
871023	2.54	24.33	15.58	57.55
871116	3.21	30.27	19.72	46.80
880327	1.52	18.99	21.71	57.78
880404	2.16	17.64	16.66	63.54
880410	3.43	22.06	15.44	59.07
880424	2.60	20.17	18.66	58.57
880507	2.55	18.53	19.69	59.23
880522	4.04	18.85	21.55	55.56
880619	3.06	18.99	13.67	64.28
880720	2.47	18.61	15.99	62.93

*contains residual toluene used in extraction of the oil from the core

Table 3: Asphaltene Agglomeration[a] for Whole Oil Sample

SAMPLE	SLOPE	STANDARD DEVIATION S.D.	Y-INTERCEPT	S.D.
880327	1.287	0.001	3.416	0.221
880404	1.211	0.014	3.381	0.165
880410	1.244	0.024	2.639	0.730
880424	1.445	0.006	-1.496	0.206
880507	1.486	0.022	2.206	0.840
880522	1.642	0.000	-2.312	0.008
880619	1.206	0.016	0.018	0.474
880720	1.431	0.013	2.722	0.346

[a] An in-house ARC method for asphaltene agglomeration using toluene as a solubilizer and pentane as a titrant.

Table 4: Analytical Data for Naphtha Cuts

SAMPLE	AROMATICS[a] (wt%)	AROMATICS[b] (vol%)	OLEFIN[b] (vol%)	NITROGEN[c] (mg/L)	SULFUR[d] (wt%)	DISTILLATION (vol%)
Core	-	-	-	<1.0	0.29	0.8*
870929	29.9	32.6	5.6	29.0	1.23	2.59
871023	35.4	34.5	11.4	137.0	1.39	2.54
871116	34.3	32.1	12.5	196.0	1.41	3.21
880327	17.2	15.7	4.1	7	0.77	1.52
880404	19.6	18.5	4.2	12	0.93	2.16
880410	34.2	31.7	6.3	17	1.16	3.43
880424	22.0	24.2	6.2	5	0.95	2.60
880507	21.8	21.9	2.9	15	1.00	2.55
880522	26.7	23.4	3.8	20	0.92	4.04
880619	16.8	15.6	3.3	13	1.03	3.06
880720	16.0	14.4	3.6	13	0.86	2.47

a measured by supercritical fluid chromatography; the balance are saturates or olefins, where present
b measured by ASTM D1319
c measured by chemiluminescence
d measured by ASTM D1552

* contains residual toluene

Table 5: Analytical Data for Middle Distillate Cuts

SAMPLE	VISCOSITY (cts)	AROMATICS[a] (wt%)	NITROGEN[b] (wt%)	SULFUR[d] (wt%)	DISTILLATION (vol%)
Core	3.99	37.5	0.0010[c]	1.63	17.7
870929	3.66	39.5	0.0075[c]	2.12	19.56
871023	3.47	48.1	0.024[c]	2.62	24.33
871116	3.69	45.4	0.028[c]	2.27	30.27
880327		36.8	0.04	2.01	18.99
880404		36.3	0.08	1.99	17.64
880410		40.6	0.05	1.73	22.06
880424		37.8	0.02	2.13	20.17
880507		37.8	0.03	1.88	18.53
880522		38.4	0.03	2.01	18.85
880619		35.4	0.05	1.97	18.99
880720		37.4	<0.01	2.01	18.61

a by supercritical fluid chromatography; the balance are saturates or olefins, where present
b measured by CHN combustion analysis unless otherwise specified
c measured by chemiluminescence
d measured by ASTM D1552

Table 6: Middle Distillate Cut Chromatographic Yields (Wt%)

SAMPLE	Fraction 1 SATURATES	Fraction 2 AROMATICS	Fraction 3 AROMATICS	Fraction 4 POLARS	Fraction 5 POLARS	Total Recovery
Core	57.5	18.3	19.2	1.0	1.5	97.5
870929	54.8	19.7	13.5	2.5	4.2	94.7
871023	45.9	18.9	30.4	1.6	1.4	98.2
871116	52.1	18.1	21.9	1.1	2.5	95.7
880327	60.4	13.1	16.3	2.0	2.5	99.3
880404	65.1	20.7	10.2	1.1	1.8	98.9
880410	52.9	17.7	26.1	1.0	1.0	98.7
880424	57.9	18.5	17.8	1.2	1.6	97.0
880507	59.5	17.2	16.9	1.9	1.1	96.6
880522	57.0	14.5	21.5	1.5	3.0	97.5
880619	60.8	17.0	16.3	1.5	1.7	97.3
880720	63.6	22.0	8.8	0.7	1.0	96.1

Table 7: Middle Distillate ^{1}H and ^{13}C NMR Data

A. Fraction 3

SAMPLE	^{1}H % ALIPHATIC	^{1}H % AROMATIC	^{13}C % ALIPHATIC	^{13}C % AROMATIC
Core	80	20	63	27
870929	75	25	59	41
871023	70	30	37	63
871116	76	24	43	57
880327	78	22	47	53
880404	77	23	47	53
880410	77	23	43	57
880424	78	22	46	54
880507	79	21	45	55
880522	82	18	49	51
880619	80	20	48	52
880720	78	22	46	54

Table 7: (cont.)

B. Fraction 4

SAMPLE	^{1}H % ALIPHATIC	^{1}H % AROMATIC	^{13}C % ALIPHATIC	^{13}C % AROMATIC
Core	96	4	78	22
870929	92	8	72	28
871023	82	18	43	57
871116	81	19	50	50
880327	88	12	62	38
880404	93	7	75[a]	25[a]
880410	88	12	56[a]	44[a]
880424	93	7	70	30
880507	88	12	49[a]	51[a]
880522	b	b	b	b
880619	93	7	78[a]	22[a]
880720	92	8	66[a]	34[a]

Table 7: (cont.)

C. Fraction 5

SAMPLE	^{1}H % ALIPHATIC	^{1}H % AROMATIC	^{13}C % ALIPHATIC	^{13}C % AROMATIC
Core	95	5	100[c]	trace
870929	96	4	86	14
871023	b	b	b	b
871116	92	8	72	28
880327	95	5	76[a]	24[a]
880404	93	7	86[a]	14[a]
880410	89	11	87[a]	13[b]
880424	91	8	70[a]	30[a]
880507	92	8	50[a]	50[a]
880522	b	b	b	b
880619	93	7	78[a]	22[a]
880720	94	6	68[a]	32[a]

[a] small sample quantity resulted in noisy spectra with relatively large uncertainty
[b] insufficient sample
[c] sample suspect

Table 8: Analytical Data for Gas Oil Cuts

SAMPLE	AROMATICS (wt%)	NITROGEN[a] (wt%)	SULFUR[c] (wt%)	DISTILLATION (vol%)
Core	8.9	0.043[b]	3.29	16.5
870929	11.3	0.072[b]	3.36	17.66
871023	56.7	0.066[b]	3.28	15.58
871116	52.2	0.10[b]	3.24	19.72
880327	56.0	0.14	3.41	21.71
880404	57.1	0.09	3.01	16.66
880410	58.4	0.10	2.86	15.44
880424	58.4	0.14	3.15	18.66
880507	58.5	0.20	3.59	19.69
880522	56.2	0.15	3.41	21.55
880619	58.1	0.10	3.46	13.67
880720	58.0	0.16	3.25	15.99

a measured by CHN combustion analysis unless otherwise stated
b measured by chemiluminescence
c measured by ASTM D1552

Table 9: Gas Oil Cut Chromatographic Yields (Wt%)

SAMPLE	Fraction 1 SATURATES	Fraction 2 AROMATICS	Fraction 3 AROMATICS	Fraction 4 POLARS	Fraction 5 POLARS	Total Recovery
Core	36.8	9.5	40.9	7.3	4.2	98.7
870929	31.7	18.9	35.4	7.9	7.9	101.8
871023	33.7	17.3	40.4	4.7	3.7	99.6
871116	35.7	8.2	37.7	6.9	9.3	97.8
880327	31.8	14.7	36.1	5.8	8.7	97.1
880404	35.1	22.9	34.8	4.2	3.9	100.9
880410	35.4	19.2	33.6	4.3	5.0	97.5
880424	34.9	19.6	32.0	4.8	6.2	97.5
880507	35.1	22.4	30.5	3.8	4.6	96.4
880522	30.6	15.8	37.3	5.9	8.0	97.6
880619	43.4	31.0	17.9	5.0	0.6	97.9
880720	39.5	25.5	22.8	3.3	5.8	96.9

Table 10: Gas Oil ^{1}H and ^{13}C NMR Data

A. Fraction 3

SAMPLE	^{1}H % ALIPHATIC	^{1}H % AROMATIC	^{13}C % ALIPHATIC	^{13}C % AROMATIC
Core	87	13	50	50
870929	84	16	54	46
871023	82	18	50	50
871116	84	16	53	47
880327	86	14	61	39
880404	83	17	52	48
880410	82	18	53	47
880424	85	15	61	39
880507	83	17	53	47
880522	85	15	53	47
880619	78	22	52	48
880720	82	18	42	58

Table 10: (cont.)

B. Fraction 5

SAMPLE	^{1}H % ALIPHATIC	% AROMATIC	^{13}C % ALIPHATIC	% AROMATIC
Core	93	7	84	16
870929	93	7	73	27
871023	85	15	51	49
871116	90	10	65	35
880327	92	8	69	31
880404	88	12	60	40
880410	90	10	68	32
880424	91	9	69	31
880507	90	10	60	40
880522	92	8	65	35
880619	88	12	68	32
880720	93	7	69	31

Table 11: Analytical Data for Residue Cuts

SAMPLE	NITROGEN[a] (wt%)	SULFUR[b] (wt%)	OXYGEN (wt%)	DISTILLATION (vol%)
Core	0.58	5.78	1.01	65.0
870929	0.51	5.53	1.03	60.19
871023	0.57	5.28	1.00	57.55
871116	0.37	5.39	1.39	46.80
880327	0.66	5.12		57.78
880404	0.58	5.17		63.54
880410	0.61	5.28		59.01
880424	0.69	5.64		58.57
880507	0.62	5.67		59.23
880522	0.67	5.63		55.56
880619	0.66	5.19		64.28
880720	0.65	5.54		62.93

a measured by CHN combustion analysis
b measured by ASTM D1552

Table 12: Residue Cut Chromatographic Yields (Wt%)

SAMPLE	Fraction 1 SATURATES	Fraction 2 AROMATICS	Fraction 3 AROMATICS	Fraction 4 POLARS	Fraction 5 POLARS	ASPHALTENES	Total Recov.
Core	11.2	4.8	19.2	11.5	30.0	23.5	100.2
870929	12.1	8.2	18.7	7.9	23.0	28.8	98.7
871023	8.8	10.3	19.7	10.2	25.6	24.1	98.7
871116	11.5	9.4	19.4	9.1	25.0	25.3	99.7
880327	10.3	7.0	17.5	32.3	2.5	25.2	94.8
880404	12.8	4.7	14.5	7.9	29.2	22.7	91.8
880410	10.2	6.9	11.6	8.5	32.5	23.8	93.5
880424	11.1	7.6	13.4	8.1	31.0	24.4	95.6
880507	10.8	6.5	12.0	8.0	29.3	24.8	91.4
880522	6.7	8.5	14.2	12.8	25.1	25.4	92.7
880619	12.2	6.5	12.5	6.6	29.8	25.4	93.0
880720	9.9	8.0	13.3	7.6	28.2	24.6	91.6

Table 13: Residue ^{1}H and ^{13}C NMR Data

A. Fraction 3

SAMPLE	^{1}H % ALIPHATIC	^{1}H % AROMATIC	^{13}C % ALIPHATIC	^{13}C % AROMATIC
Core	92	8	67	33
870929	91	9	65	35
871023	93	7	70	30
871116	92	8	72	28
880327	92	8	65	35
880404	92	8	72	28
880410	93	7	68	32
880424	92	8	66	34
880507	91	9	72	28
880522	93	7	67	33
880619	92	8	72	28
880720	92	8	73	27

Table 13:

B. Fraction 5

SAMPLE	^{1}H % ALIPHATIC	% AROMATIC	^{13}C % ALIPHATIC	% AROMATIC
Core	94	6	68	32
870929	92	8	66	34
871023	92	8	64	36
871116	92	8	68	32
880327	92	8	67	33
880404	92	8	67	33
880410	93	7	70	30
880424	93	7	68	32
880507	92	8	68	32
880522	94	6	68	32
880619	93	7	70	30
880720	93	7	71	29

PART II

INTRODUCTION

The report for Part I of this project presented the results for the oils produced from well EX-32 over a one year period by the in situ combustion pilot at Wolf Lake, Alberta. A detailed set of chemical analyses indicated that the oils produced by the fireflood process were upgraded relative to a pre-fireflood core sample, with a reduction in density and viscosity coupled to increases in the amount of material boiling in the naphtha and middle distillate ranges. In addition, there were shifts to lower carbon number in some of the compound classes of the produced oils versus the core. The chemical composition of the naphtha distillation cut, in particular, underwent considerable change as a result of the combustion process. Significant amounts of C_6 to C_{12} n-paraffins (from thermal cracking and distillation near the firefront) were found in the naphtha cuts from the produced oils but were absent in the core sample. The lack of an increase in asphaltenes or heteroatom content during the portion of fireflood cycle studied was an auspicious sign for the subsequent upgrading of the produced oil.

For the final phase of this project, it was agreed to examine a number of samples produced from well EX-6 over an eight month period. This well was chosen for several reasons. Since it is at the opposite end of the field to EX-32, the effects of the in situ combustion process on the produced oils from different areas of the field could be compared. Secondly, it was understood that EX-6 exhibited a relatively high production rate over the full period of

the combustion pilot. Finally, it was also understood that EX-6 was connected to an injector utilizing a different strategy than that linked to EX-32. Eight production samples from EX-6 were available spanning the period from 02/11/87 to 16/09/88.

In previous discussions with BP representatives, the following three factors were identified as being important:

1. The extent of low temperature oxidation (LTO) in the produced oil and the chemical and physical factors which are correlated with these LTO products.

2. The relationship between emulsification of the produced fluids and the properties of the oil phase.

3. The effects of the in situ combustion process on the subsequent upgradability of the produced oil.

Based on the results from the previous phase of the project, it was felt that the following analyses would provide the most useful information with respect to these three factors:

a) elemental analysis (C,H,N,O,S)

b) distillation yields

c) asphaltene content

d) acid number

e) density

f) viscosity

An examination of these data revealed anomalous behaviour for some of the produced oils and four such samples were chosen for more detailed analyses. These four samples were distilled into four fractions which were subsequently separated into general compound classes by column chromatography. Some of these chromatographic fractions were analyzed by gas chromatography - mass spectrometry (GC-MS) to examine changes in carbon number and individual compound class distributions. A flow scheme which outlines the sample separation protocol and analytical techniques employed is shown in Figure 1. The results of these analyses and their relationship to the three factors stated above are discussed in the report. In addition, comparisons between the results for the EX-6 produced oils and the core sample data are made.

EXPERIMENTAL

All production samples were collected from the drilled well EX-6 (five spot pattern) at the BP Wolf Lake lease in the Cold Lake oil sands deposit. These samples were labelled with the dates they were collected (year/month/day e.g. 880331); the fireflood commenced on approximately September 1, 1987. Previous to this, a core sample was obtained from the same field. The bitumen from the core sample was isolated by toluene extraction. To minimize the loss of light ends during subsequent rotary evaporation, 0.5% residual toluene was deliberately left in the extract. Water and solids

were removed from the production samples by ultracentrifugation prior to analysis.

The density of each whole oil was determined at 25 °C using an Anton Parr Model DMA60 density meter with a DMA601 measuring tube capable of measuring to five decimal places. The instrument was thermostated using a recirculating temperature bath with a control accuracy of ±0.01 °C. The temperature was monitored to the nearest 0.1 °C.

The viscosity measurements were performed on a Brookfield Viscometer model RVT equipped with a cone and plate measuring head. The viscometer was maintained at 25 °C using a temperature bath capable of controlling to ±0.2 °C at the viscometer head.

The %water analyses were performed on a Mitsubishi Model CA-02 Karl Fischer instrument.

The carbon, hydrogen, nitrogen, and oxygen analyses for the whole oil samples were determined on a Perkin-Elmer 240 Elemental Combustion Analyzer. The wt% sulfur in the oils was determined by the ASTM D1552 (combustion) procedure.

The acid numbers (total) were measured by the ASTM D664 method using a Metrohm Model 636 automatic titrator with a glass and Ag/AgCl reference electrode pair.

Simulated distillation on the whole oil samples was carried out using the ASTM D-2 proposed P167 method run on a Hewlett Packard 5890 GC equipped with auto injection.

The time slice data was collected on an HP3392A integrator and transferred to a VAX 11/750 computer for data processing.

The four selected whole oil samples were distilled into four distillation cuts: naphtha (115-204 °C), middle distillate (204-345 °C), gas oil (345-442 °C), and residue (>442 °C) cuts. These were produced using a Nester Faust Spinning Band Distillation system which consisted of a 480 mm x 7 mm column with a Monel band spinning at approximately 29 rev/s. The distillations were conducted at a pressure of 5.0 mm Hg and a reflux rate of 5:1. All temperatures were converted to atmospheric equivalent temperatures (AET) using the Maxwell-Bonnell equation.

The asphaltenes from each residue distillation cut were separated by n-pentane precipitation. A 2-5 gram sample of the residue was dissolved in an equal amount of toluene to which n-pentane (40 volumes) was added. The precipitated asphaltenes were collected on a 0.45 μm frit and weighed after drying.

The column chromatographic separations by compound class (saturates, aromatics, polars, and asphaltenes or SAPA) of the middle distillate, gas oil, and residue distillation cuts were performed using a modification of the method of Sawatzky [13]. A weighed 2 mL sample of a middle distillate or gas oil cut (or a sample of maltenes from a deasphalted residue cut) was separated on a silica/alumina column using the following solvent system. Fraction 1 (saturates) was collected with 150 mL of pentane followed by fraction 2 (aromatics 1: mainly monoaromatics), which was eluted with 200 mL of pentane containing 5% toluene. Fraction 3 (aromatics 2: mostly di-aromatics) was

collected with 300 mL of 30% toluene in pentane while fraction 4 (polars 1) was eluted with 300 mL of toluene/pentane (70/30, v/v). The remaining polar fraction 5 (polars 2) was eluted with 300 mL of methylene chloride/methanol (50/50, v/v). The chromatographic solvents were removed by evaporation in a fumehood. Any residual solvent in the polar fractions was removed by heating in an oven at 100 °C for 10 min. The total recoveries from the chromatographic separation of some of the gas oil cuts were lower than expected (92-97%). This was attributed to varying amounts of a dark, polar material which did not elute from the chromatographic adsorbent. The low recoveries from some of the middle distillate cuts were due to loss of light ends during solvent evaporation.

The gas chromatography-mass spectrometry (GC-MS) analyses were carried out on at Finnigan Mat 4500 Gas Chromatograph - Mass Spectrometer. An Al-clad SGE HT-5 or a Hewlett Packard P.N.A. capillary column was used for the gas chromatographic separations. The HT-5 column was 25 m x 0.22 mm I.D. with a 0.1μm film thickness. The P.N.A. column was 30 m x 0.25 mm I.D. with a 0.1μ film thickness. A split or splitless injection system was used for sample introduction on both capillary columns. For the HT-5 columns the gas chromatography was performed isothermally at 50 °C for 5 min followed by temperature programming at 10 °C per min. The maximum temperature for middle distillate and gas oil fractions was 320 °C while residue fractions were run up to 430 °C. For the P.N.A. column (naphtha cuts), the chromatography was performed isothermally at 35 °C for 10 min followed by two temperature programs: 3 °C per min to 150 °C then 10 °C per min to 200 °C.

The mass spectra of the gas chromatographic effluents were obtained using nitrous oxide (N_2O) chemical ionization (CI). The scan rate was generally 0.95 sec to scan from 33 to 500 amu. The CI spectra were obtained using a Townsend discharge ionization source with N_2O at approximately 0.45 torr at 120 °C. The spectra were summed over the length of the chromatogram to give a picture of the types of chemical structures found in these samples. The response of the mass spectrometer detector for different molecular weight components was not determined due to a lack of suitable standard compounds. It is generally recognized that the response drops off with increasing molecular weight and thus the data presented are not an accurate representation of the relative concentrations of compounds within a particular sample. The data do, however, accurately represent the differences between samples and do give a semi-quantitative picture of the distribution of compound types within a given sample.

RESULTS AND DISCUSSION

Some analytical data for the core sample and the produced oils from EX-6 are shown in Table 1. The viscosity reduction found for the fireflood produced oils in the previous phase of this contract (i.e. from EX-32) versus the core sample is also observed for the EX-6 samples, although to a lesser degree. There was also a slight decrease in density for the EX-6 oils relative to the core sample. This viscosity and density data indicate that the produced oil from EX-6 was upgraded with respect to the core sample but to a lesser extent than the EX-32 produced oils. There are no obvious trends in the

variations of wt% carbon, hydrogen, or sulfur between the core and produced EX-6 samples or among the produced oils themselves. Neither is there any apparent trend in the calculated H/C atomic ratios (not shown) which range from 1.47 to 1.56. All but one of the EX-6 oils produced by the in situ combustion process have lower nitrogen and sulfur contents than the core sample. This behaviour, which is clearly an advantage in any subsequent upgrading process, was also found for the EX-32 fireflood oils. The small variations in wt% oxygen for the core and EX-6 oils exhibit no observable pattern and there appears to be no correlation between wt% oxygen and acid number. This result was in contrast to that found for the EX-32 samples, where a small increase in wt% oxygen between the core and early production samples was accompanied by a decrease in acid number. The oxygen content and acid number data for the core and EX-6 oils indicate that, if any low temperature oxidation (LTO) is occurring as a result of the in situ combustion process, the LTO products do not appear in the produced oil. The heteroatom content and acid number data for the core and EX-6 produced oil samples imply that there is no chemical basis (such as an increase in hydrocarbon-based surfactants) to rationalize the "light" emulsions which these fireflood-produced oils formed. Overall, the data in Table 1 suggest that the produced oils from EX-6 are affected less by the in situ combustion process than the EX-32 oils.

Some simulated distillation data for the core and EX-6 samples are shown in Table 2. These results indicate that the fireflood produced oils were all lighter than the core sample, which required a higher temperature to acheive 5, 20, and 40% off and only yielded 44.2% distillate at the high temperature limit (equivalent) of the technique. The lighter nature of the produced oils with respect to distillation is consistent with the

reduced viscosity of these oils relative to the core sample.

In Figures 2 and 3, respectively, the change in viscosity and density for the EX-6 produced oils are plotted versus a chronological ordering of the samples. Also plotted in this fashion (Figure 4) is the temperature required to distill 5% of the produced oils, based on the simulated distillation data. These three plots were prepared to identify any samples from within the eight EX-6 fireflood oils which exhibited anomalous behaviour and warranted a more detailed characterization. Based on the fact that each of these plots exhibit an early maximum and a later minimum, two samples bracketing the maximum (871102, 880331) and two around the minimum (880608, 880701) were chosen for further analysis.

These four samples were separated into four distillation cuts by spinning band distillation: naphtha (IBP-204 °C), middle distillate (204-345 °C), gas oil (345-442 °C), and residue (>442 °C). The normalized yields (wt%) for the four EX-6 production samples and the core sample are shown in Table 3, with recoveries in the range 99.1-99.6%. Also shown in Table 3 are the asphaltene contents of the samples as a wt% of the whole oil. It may be observed that the naphtha contents of the samples produced by in situ combustion are 3-7 times larger than that of the core sample. This behaviour may be compared to the corresponding increase of 5-13 times for the EX-32 fireflood samples over the core sample. These results indicate that the dilution of the produced oils from EX-6 by light material from near the firefront is less than for the corresponding EX-32 oils. A comparison of the distillation yields for the middle distillate cuts of EX-6 and EX-32 is in accord with this conclusion. The early produced oil samples from EX-32

contain up to 30 wt% middle distillate versus 17.7 wt% for the core sample while the middle distillate contents of the core sample and the EX-6 produced oils show little variation. The wt% asphaltenes in the produced EX-6 oils exhibit a modest but consistent decrease relative to the core sample. This behaviour is in contrast to that of the fireflood produced EX-32 samples which, on average, contained slightly more asphaltenes than the core sample. Overall, the distillation data indicate that the EX-6 oils produced by the in situ combustion process are upgraded relative to the core sample although to a lesser extent than the analogous EX-32 samples.

The middle distillate, gas oil, and residue distillation cuts from the core and four EX-6 produced oils were each separated into five fractions by a column chromatographic technique. The residue cut was deasphalted before column chromatography. The results of this fractionation are shown in Table 4. All of the middle distillate cuts contain >50% saturates with aromatics 1 (mostly mono-aromatics) and aromatics 2 (predominantly di-aromatics) making up most of the balance. The split between aromatics 1 and 2 is nearly equal for the core sample but heavily favours aromatics 2 for the EX-6 produced oils. The gas oil cuts exhibit a more even distribution between saturates and aromatics fractions, with the total aromatics constituting the largest portion for all the samples. The aromatics component in the core and early fireflood samples is dominated by di-aromatics while the later EX-6 samples have a more even distribution between aromatics 1 and 2. The amount of polars in the gas oil cuts is approximately twice that of the middle distillate cuts. For the residue distillation cuts, the total polars make up >50% of the deasphalted samples, with polars 2 making the major contribution. This trend to increased polars content with increasing boiling point of distillation cut is

expected behaviour. There are also significant amounts of aromatics present in the residue samples, with the distribution weighted heavily towards di-aromatics (aromatics 2). Within each of the three distillation cuts, there do not appear to be any obvious trends between the core sample and the oils produced by in situ combustion or within the produced EX-6 samples themselves.

A gas chromatography - mass spectrometry (GC-MS) analysis of some of the column chromatographic fractions was used to examine detailed changes in the chemical composition of the EX-6 produced oils. Based on the results obtained during the previous phase of this project (i.e. EX-32), only the saturates, aromatics 1, and aromatics 2 fractions were analyzed by GC-MS. In addition to these chromatographic fractions, the whole naphtha distillation cuts were also analyzed by GC-MS. The GC-MS data for the naphtha distillation cuts from the EX-6 produced oils are shown in Figure 5, which also includes the corresponding data from the core sample. This data is presented in bargraph form, with a breakdown both by detailed chemical compound class and carbon number distribution within these classes. With the exception of a relative reduction in the amount of the alkyl benzene compound class in the EX-6 samples versus the core, there is little change in the compound class distribution within the naphtha cuts as a result of the in situ combustion process. The carbon number distributions of the compound classes in the produced EX-6 oils exhibit a small but consistent shift towards heavier compounds, relative to the core sample. This result is the opposite of that observed for the EX-32 oils. This behaviour indicates that dilution of the produced oil by light material either distilled from near the firefront or produced by thermal cracking at the firefront (as observed for the EX-32 oils) did not occur to any

significant extent for the EX-6 samples.

A bargraph representation of the GC-MS data for fraction 1 (saturates) from the middle distillate cuts of the EX-6 produced oils is shown in Figure 6, again with the corresponding data for the core sample included. It may be observed that there are only minor changes in the compound class distributions between the core and produced oil samples or within the latter. As was observed from the naphtha GC-MS data (Figure 5), the carbon number distributions of the compound classes in the middle distillate saturate fraction of the EX-6 oils exhibit a small but consistent shift towards higher carbon number, relative to the core sample.

Most of the important changes in the compound class and carbon number distributions for the EX-32 produced oils were reflected in the GC-MS data for the naphtha distillation cut and the chromatographic fractions from the middle distillate cut. It was felt that the lack of significant changes in the corresponding data for the EX-6 samples did not warrant the costly and time-consuming preparation of the remaining GC-MS data into the bargraph format. Thus, a tabular format was used for the rest of this data. The GC-MS data for fractions 2 and 3 from the middle distillate cut are shown in Tables 5 and 6, respectively, as relative intensity by compound class and carbon number. The corresponding data for fractions 1, 2, and 3 from the gas oil and residue distillation cuts are shown in Tables 7-9 and 10-12, respectively.

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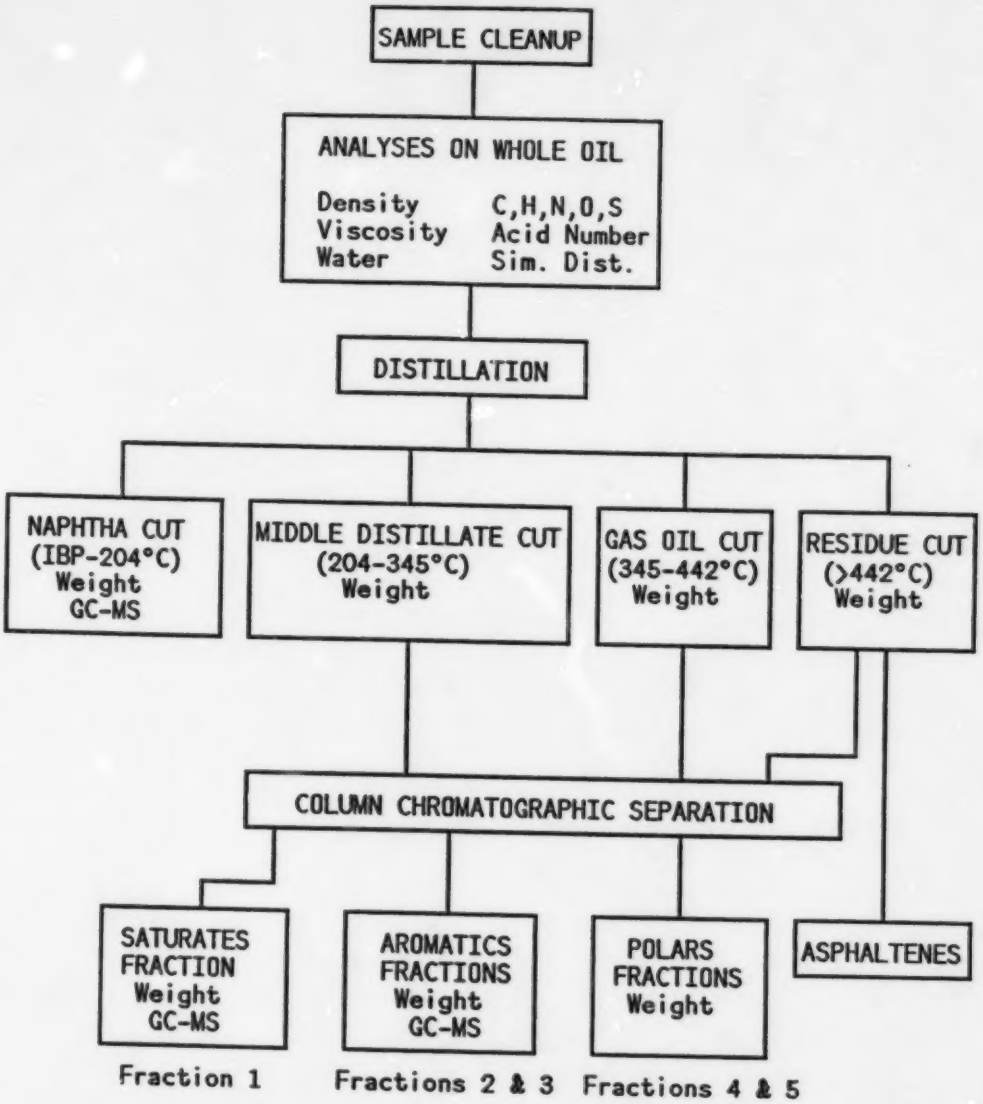


Figure 1. Flow scheme showing sample separation procedure and analytical techniques.

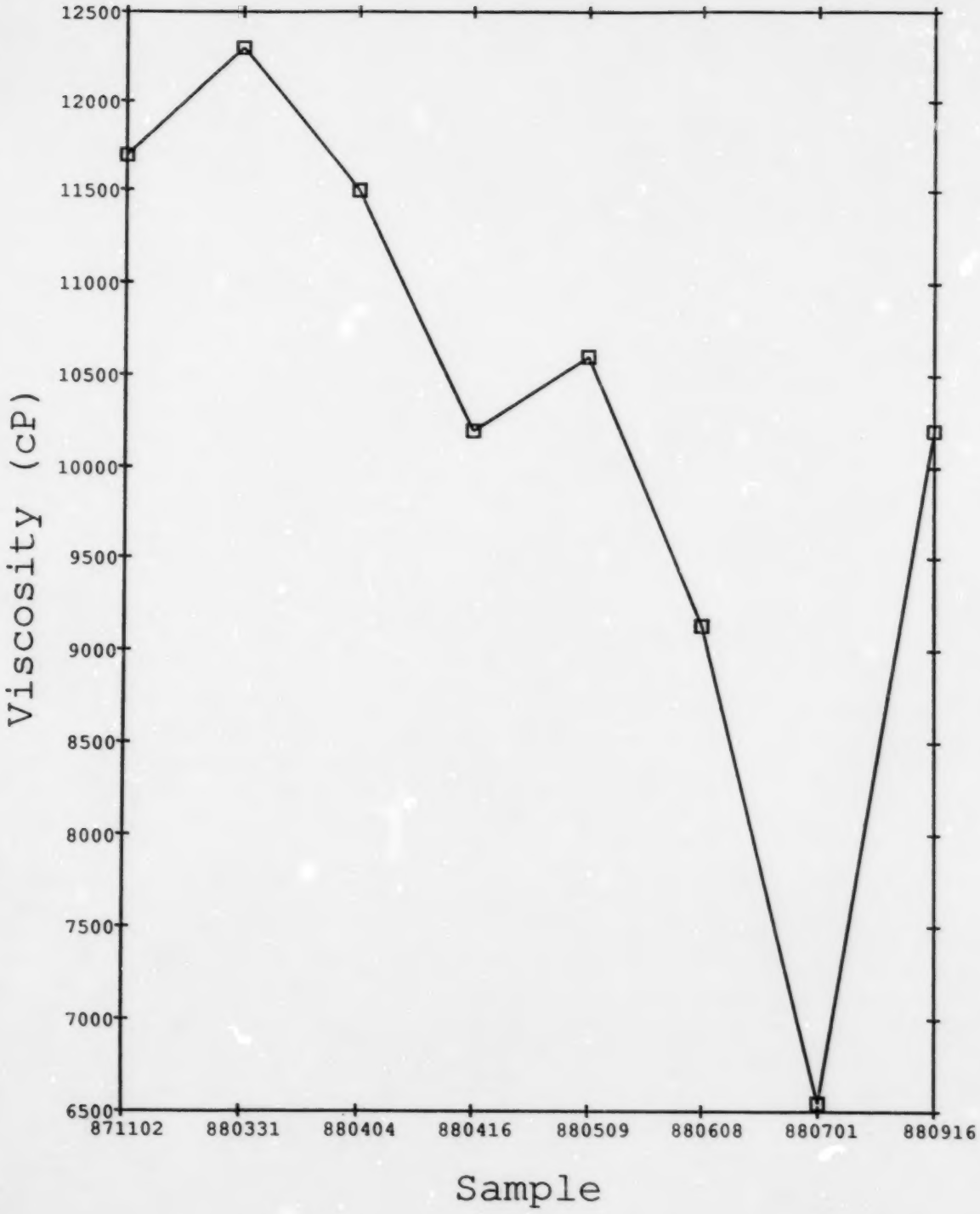


Figure 2. A plot of viscosity versus a chronological ordering of the EX-6 produced oil samples.

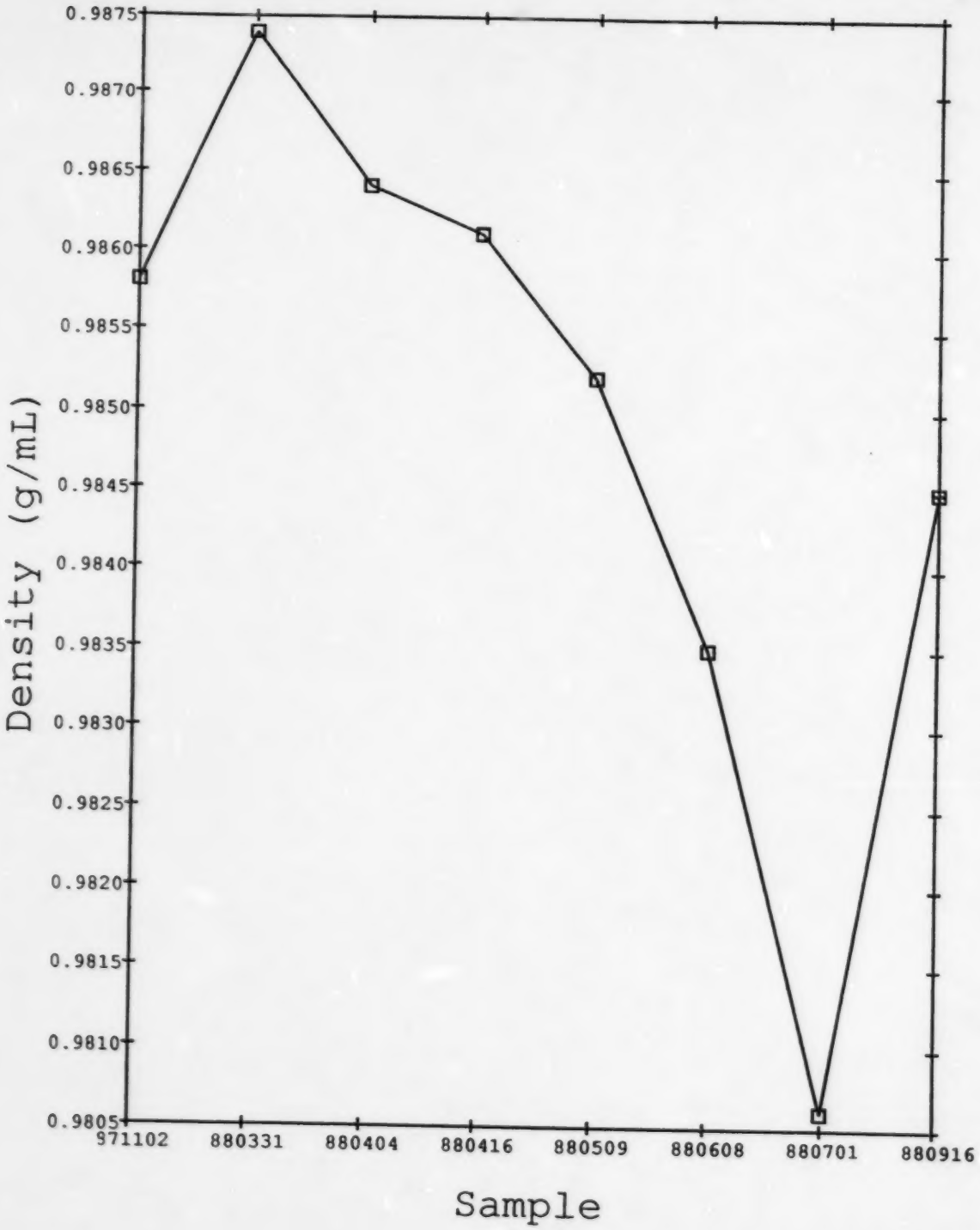


Figure 3. A plot of density versus a chronological ordering of the EX-6 produced oil samples.

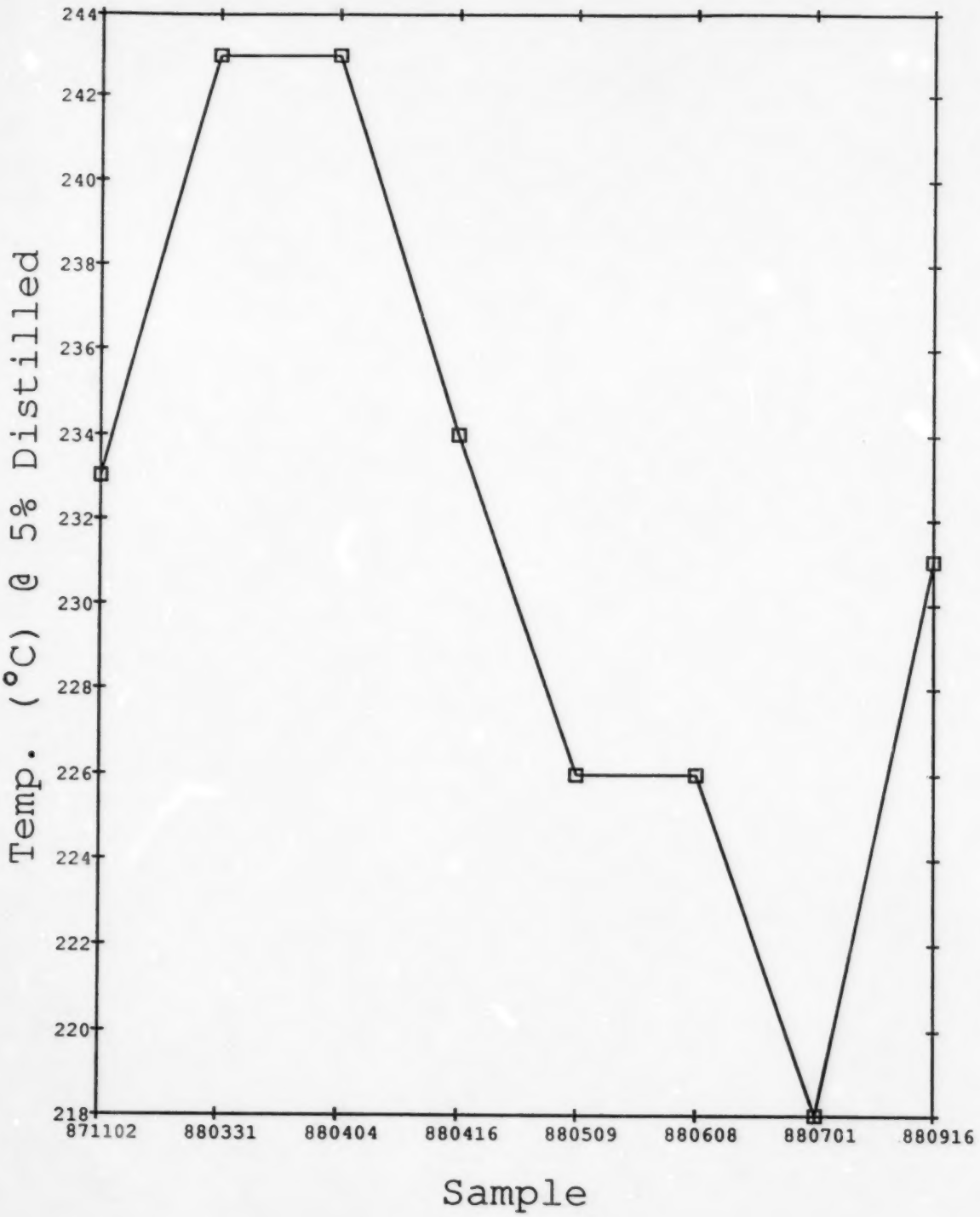


Figure 4. A plot of temperature required to distill 5% (from simulated distillation data) versus a chronological ordering of the EX-6 produced oil samples.

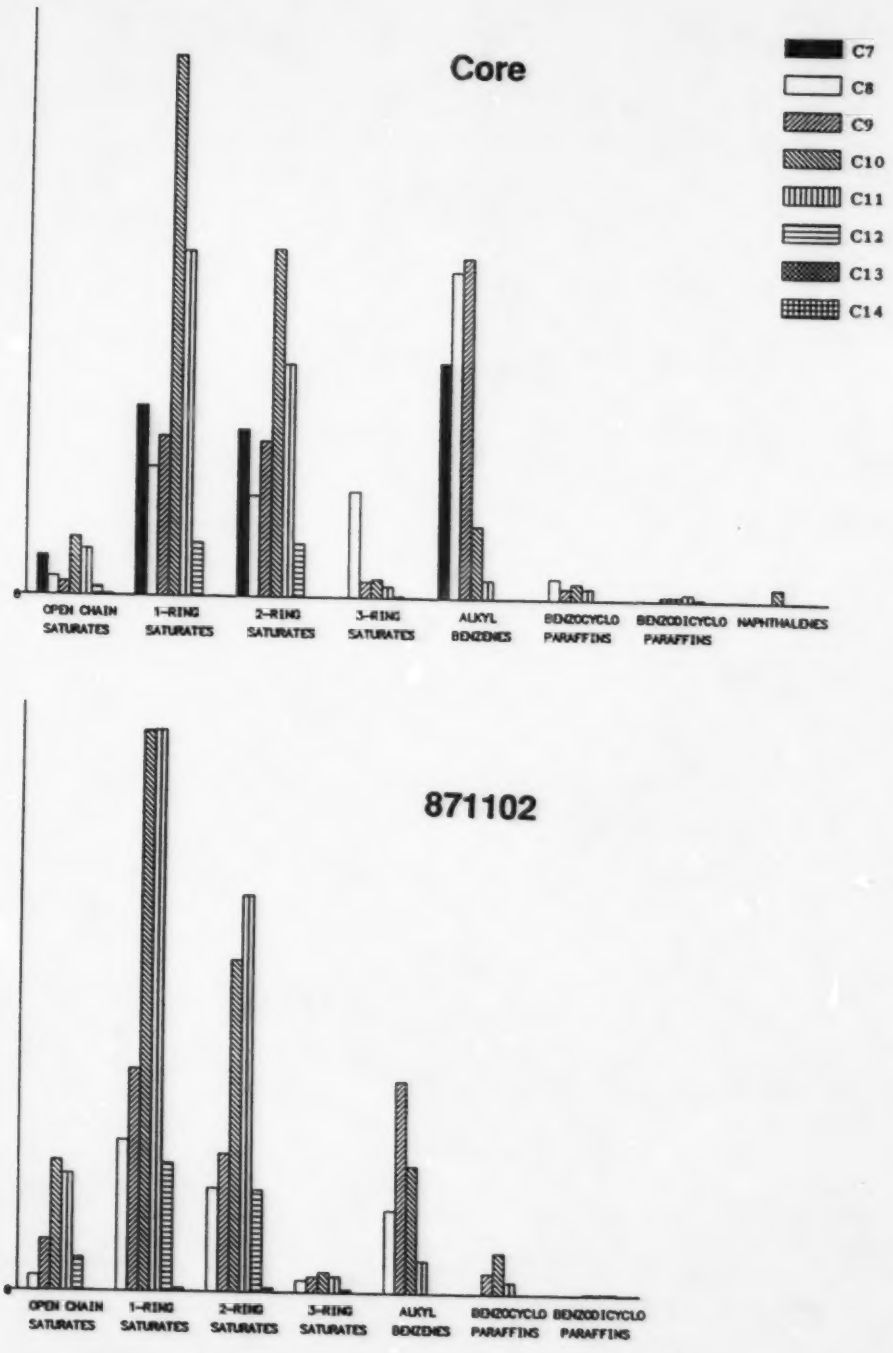
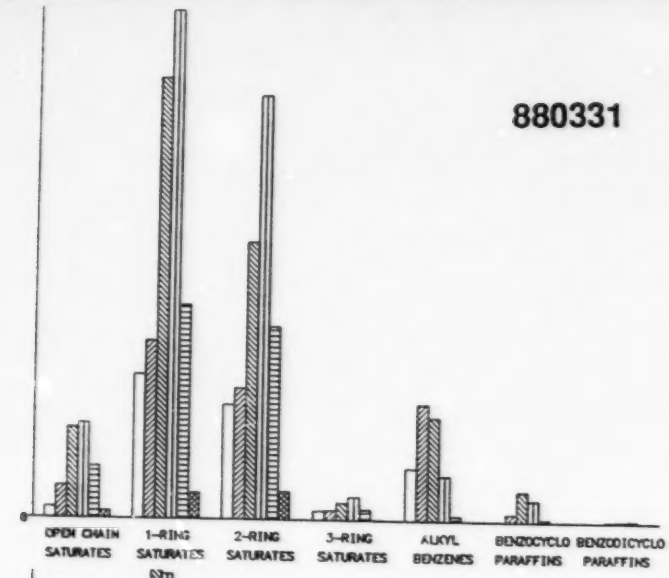
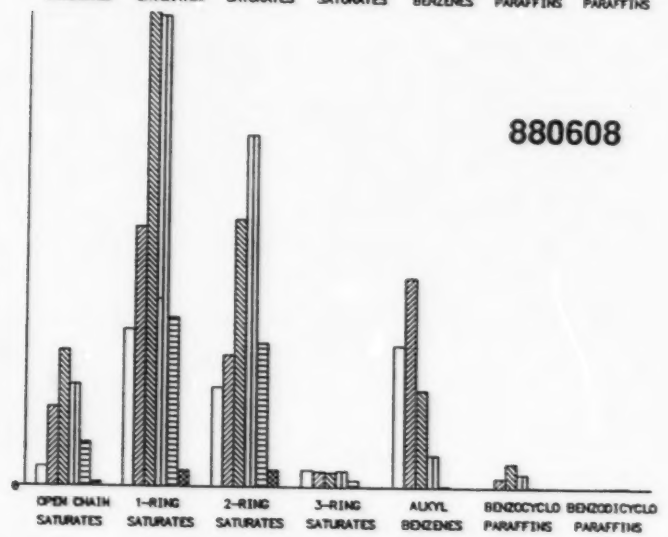


Figure 5. A bargraph representation of the GC-MS data for the naphtha distillation cuts of the core and selected EX-6 produced oils.





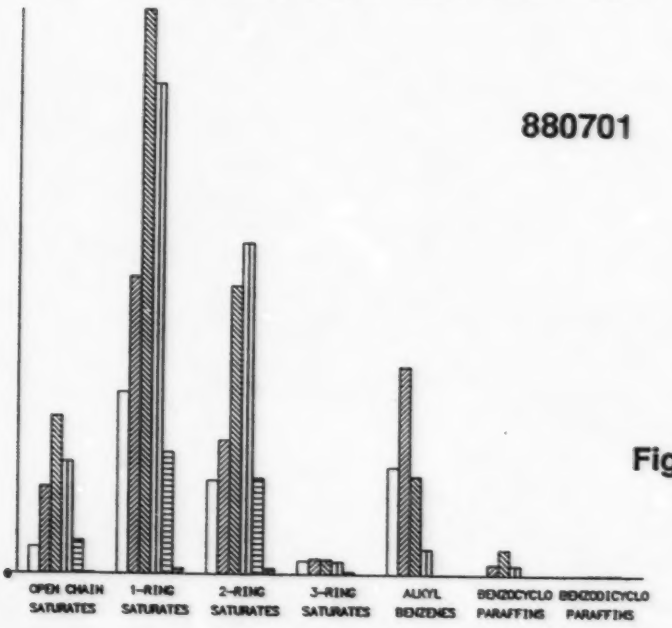


Figure 5. (Cont.)

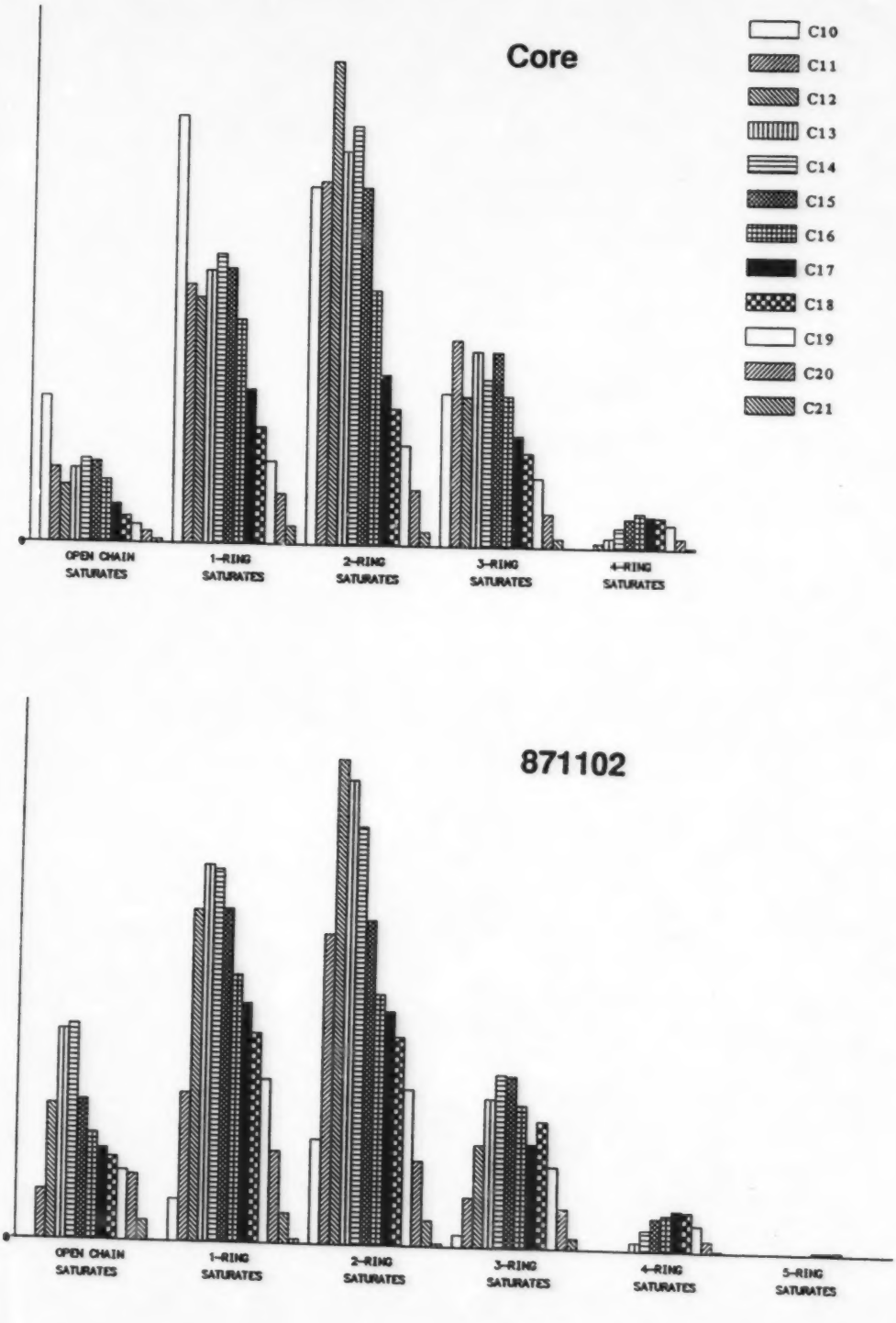
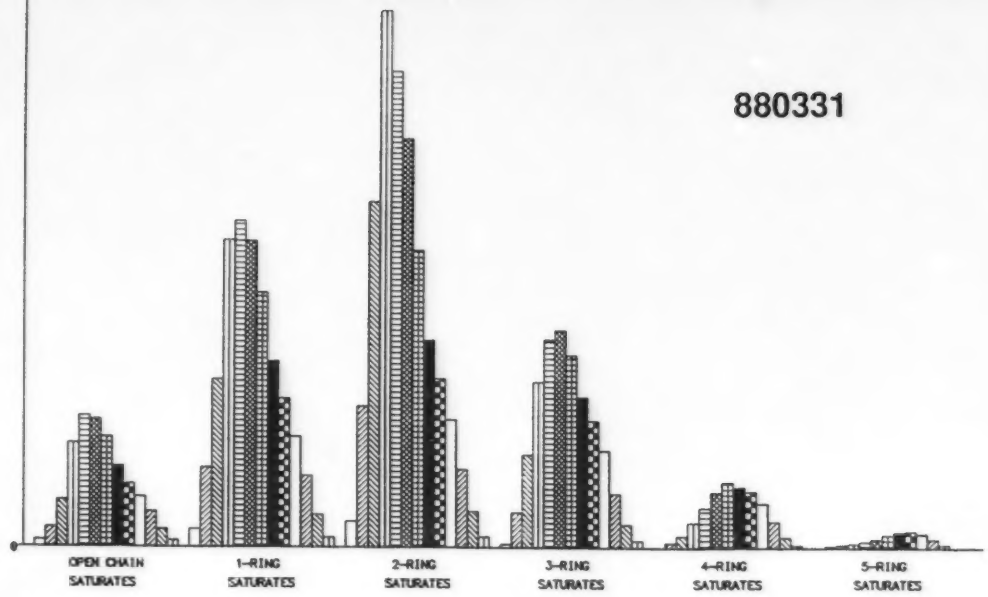
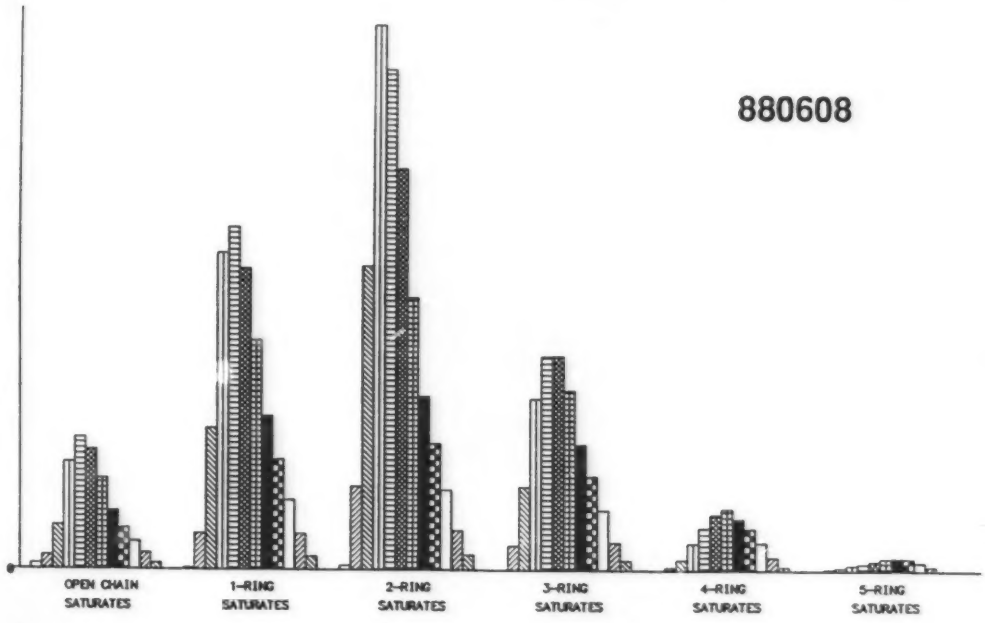


Figure 6. A bargraph representation of the GC-MS data for the saturate fraction from the middle distillate cuts of the core and selected EX-6 produced oils.





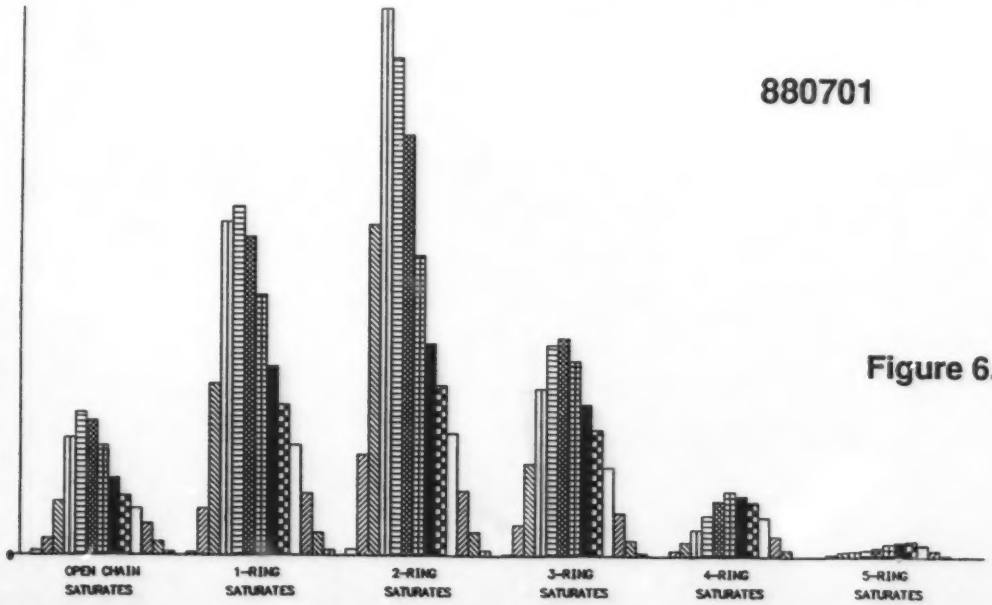


Figure 6. (Cont.)

Table 1. Analytical Data for Whole Oil Samples

Sample	Density (g/mL, 25°C)	Viscosity (cP, 25°C)	Water (wt%)	Carbon (wt%)	Hydrogen (wt%)	Nitrogen (wt%)	Oxygen (wt%)	Sulfur (wt%)	Acid No. (mg KOH/g)
Core	0.9894	49,400		83.90	10.67	0.60	0.47	4.8	1.32
871102	0.9858	11,700	0.77	83.90	10.35	0.34	0.46	4.7	2.35
880331	0.9874	12,300	0.25	83.65	10.42	0.38	0.61	4.6	2.00
880404	0.9864	11,500	0.33	83.79	10.56	0.49	0.59	4.7	1.47
880416	0.9861	10,200	0.19	83.80	10.52	0.37	0.42	4.7	1.46
880509	0.9852	10,600	0.31	83.52	10.74	0.44	0.35	4.7	1.48
880608	0.9835	9,140	0.19	83.98	10.33	0.37	0.40	5.0	1.03
880701	0.9806	6,540	0.20	83.10	10.87	0.41	0.42	4.4	1.13
880916	0.9845	10,200	0.20	83.69	10.94	0.35	0.45	4.7	2.16

Table 2. Simulated Distillation Data for Whole Oils

Sample	5% Off (°C)	20% Off (°C)	40% Off (°C)	% Distilled (at 512 °C)
Core	254	357	472	44.2
871102	233	336	454	48.0
880331	243	340	452	48.5
880404	243	341	454	48.0
880416	234	339	455	47.8
880509	226	339	461	46.7
880608	226	339	462	46.6
880701	218	325	445	49.4
880916	231	337	457	47.5

Table 3. Distillation Data and Asphaltene Yields (Wt%)

Sample	Naphtha (IBP-204°C)	Mid-Distillate (204-345°C)	Gas Oil (345-442°C)	Residue (>442°C)	Asphaltenes*
Core	0.3	17.7	16.5	65.5	15.4
871102	1.4	18.0	14.9	65.7	15.1
880331	1.2	17.4	17.2	64.2	15.0
880608	2.1	15.4	15.0	67.5	14.5
880701	2.1	17.2	17.1	63.6	14.4

* as wt% of whole oil

Table 4. Chromatographic Yields from Distillation Cuts

Sample	Distillation Cut (°C)	Chromatographic Fractions					Recovery (wt%)
		Saturates (wt%)	Aromatics 1 (wt%)	Aromatics 2 (wt%)	Polars 1 (wt%)	Polars 2 (wt%)	
Core	204-345	57.5	18.3	19.2	1.0	1.5	97.5
871102	204-345	55.9	2.6	32.0	3.3	0.0	94.0
880331	204-345	65.1	1.6	31.1	3.4	0.0	101.2
880608	204-345	62.5	3.5	28.3	3.0	0.0	97.6
880701	204-345	62.1	2.6	30.9	2.6	0.0	98.2
Core	345-442	36.8	9.5	40.9	7.3	4.2	98.7
871102	345-442	40.5	6.7	38.6	6.6	0.0	92.4
880331	345-442	44.5	7.3	41.5	7.0	0.0	100.3
880608	345-442	36.5	23.6	28.3	5.2	0.0	96.9
880701	345-442	35.3	13.7	30.9	5.4	0.0	100.8
Core	>442	14.7	6.3	25.0	15.1	39.1	100.2
871102	>442	11.9	11.3	23.1	12.7	38.0	97.0
880331	>442	13.6	2.7	25.4	8.4	49.7	99.8
880608	>442	16.3	4.9	19.9	9.9	48.8	99.8
880701	>442	12.5	6.9	20.7	7.5	49.2	96.8

871102

0 TYPE OF RING	1 C9	2 C10	3 C11	4 C12	5 C13	6 C14	7 C15	8 C16	9 C17
1 ALKYLBENZENES	61.5	100.0	66.0	46.4	43.5	48.0	49.8	49.7	45.6
2 BENZOCYCLOPARAFFINS		31.5	40.0	36.9	29.6	28.9	32.2	35.9	35.5
3 BENZODICYCLOPARAFFINS		23.4	19.3	12.4	8.8	7.2	7.9	9.9	11.9
4 NAPTHALENES		25.6	17.0	6.9	4.9	3.2	2.1	1.7	1.6
5 NAPTHOCYCLOPARAFFINS		40.3	20.1	13.3	11.4	9.9	6.8	4.9	3.5
6 FLOURENES		30.4	17.2	13.0	12.2	11.7	8.4	6.2	4.8
7 PHENANTHRENES		18.9	16.8	14.4	13.0	13.1	12.8	13.2	12.4
8									

0 TYPE OF RING	10 C18	11 C19	12 C20	13 C21	14 C22	15 C23
1 ALKYLBENZENES	39.9	31.6	21.1	9.3	2.6	0.4
2 BENZOCYCLOPARAFFINS	36.8	28.7	19.2	9.2	3.0	0.5
3 BENZODICYCLOPARAFFINS	13.6	12.5	9.4	4.7	1.5	0.2
4 NAPTHALENES	2.3	2.8	2.6	1.7	0.5	0.1
5 NAPTHOCYCLOPARAFFINS	3.6	3.7	3.6	3.2	2.0	0.7
6 FLOURENES	6.0	5.2	5.0	4.7	3.2	1.2
7 PHENANTHRENES	11.1	10.1	8.1	5.3	2.2	0.5
8						

Table 5. GC-MS data for fraction 2 from the middle distillate cuts of selected EX-6 produced oils.

880331

0 TYPE OF RING	1 C14	2 C15	3 C16	4 C17	5 C18	6 C19	7 C20	8 C21	9 C22
1 ALKYLBENZENES	59.9	72.1	90.6	100.0	97.8	87.8	67.5	34.8	11.6
2 BENZOCYCLOPARAFFINS	63.4	54.0	68.3	76.7	90.4	80.0	62.5	35.0	13.9
3 BENZODICYCLOPARAFFINS	19.4	20.9	22.2	24.2	31.0	34.6	33.1	20.8	8.4
4 NAPTHALENES	7.3	6.1	6.0	6.3	7.1	8.7	9.9	7.6	3.8
5 NAPTHOCYCLOPARAFFINS		19.0	15.2	12.6	14.6	16.4	16.2	15.0	10.8
6 FLOURENES		13.6	13.8	12.2	22.1	21.0	21.1	20.8	15.8
7 PHENANTHRENES		14.0	18.5	23.6	25.4	24.8	22.1	17.4	8.8
8									

0 TYPE OF RING	10 C23	11 C24	12
1 ALKYLBENZENES	2.7	0.4	
2 BENZOCYCLOPARAFFINS	3.5	0.5	
3 BENZODICYCLOPARAFFINS	2.1	0.3	
4 NAPTHALENES	1.2	0.2	
5 NAPTHOCYCLOPARAFFINS	5.0	1.3	
6 FLOURENES	7.1	1.6	
7 PHENANTHRENES	2.9	0.7	
8			

Table 5. (Cont.)

880608

0 TYPE OF RING	1 C14	2 C15	3 C16	4 C17	5 C18	6 C19	7 C20	8 C21	9 C22
1 ALKYLBENZENES	52.0	75.8	99.5	100.0	83.7	58.3	33.5	13.0	3.6
2 BENZOCYCLOPARAFFINS	47.8	58.8	77.7	82.7	80.1	57.4	34.0	14.7	4.7
3 BENZODICYCLOPARAFFINS	18.5	17.8	22.7	28.6	31.8	27.8	19.3	9.1	3.0
4 NAPTHALENES	4.9	4.9	5.0	5.5	6.6	7.3	6.4	3.7	1.4
5 NAPTHOCYCLOPARAFFINS		7.1	5.6	5.1	6.6	6.8	6.0	4.6	2.6
6 FLOURENES		5.7	5.8	6.9	13.1	9.9	8.6	7.3	4.6
7 PHENANTHRENES		11.2	16.8	20.4	21.7	19.0	13.8	8.2	3.1
8									

0 TYPE OF RING	10 C23	11 C24
1 ALKYLBENZENES	0.8	0.1
2 BENZOCYCLOPARAFFINS	1.1	0.2
3 BENZODICYCLOPARAFFINS	0.7	0.1
4 NAPTHALENES	0.3	0.0
5 NAPTHOCYCLOPARAFFINS	1.0	0.2
6 FLOURENES	1.7	0.3
7 PHENANTHRENES	0.8	0.1
8		

Table 5. (Cont.)

880701

0 TYPE OF RING	1 C9	2 C10	3 C11	4 C12	5 C13	6 C14	7 C15	8 C16	9 C17
1 ALKYLBENZENES	61.2	100.0	63.0	44.5	50.8	53.2	55.4	50.5	40.3
2 BENZOCYCLOPARAFFINS		47.9	62.0	57.5	53.8	61.8	59.9	53.4	42.6
3 BENZODICYCLOPARAFFINS		27.0	24.1	18.6	16.3	19.2	23.3	24.5	22.4
4 NAPTHALENES		23.9	17.6	9.2	5.6	3.4	3.0	3.4	4.0
5 NAPHTHOCYCLOPARAFFINS		25.2	11.7	7.8	5.9	4.6	2.9	2.5	2.6
6 FLOURENES		16.6	9.1	6.6	5.8	5.4	3.9	3.0	3.6
7 PHENANTHRENES		15.1	11.7	10.3	10.2	11.6	12.4	11.8	11.4
8									

0 TYPE OF RING	10 C18	11 C19	12 C20	13 C21	14 C22
1 ALKYLBENZENES	30.4	18.7	8.5	2.3	0.3
2 BENZOCYCLOPARAFFINS	33.9	19.2	8.6	2.7	0.4
3 BENZODICYCLOPARAFFINS	18.3	10.8	4.7	1.3	0.1
4 NAPTHALENES	4.1	3.0	1.5	0.4	0.4
5 NAPHTHOCYCLOPARAFFINS	2.7	2.3	1.7	1.1	0.8
6 FLOURENES	4.4	3.2	2.6	1.9	0.1
7 PHENANTHRENES	9.4	7.3	4.5	1.9	0.4
8					

Table 5. (Cont.)

871102 S

0 TYPE OF RING	1 C10	2 C11	3 C12	4 C13	5 C14	6 C15	7 C16	8 C17	9 C18
1 ALKYLBENZENES	31.9	31.5	47.2	67.7	71.5	71.8	63.4	50.9	37.9
2 BENZOCYCLOPARAFFINS	29.5	46.0	70.9	78.9	91.5	79.1	63.7	50.1	38.9
3 BENZODICYCLOPARAFFINS	21.6	32.6	24.2	32.2	38.9	47.7	47.3	40.2	31.9
4 NAPTHALENES	60.4	55.2	83.5	100.0	70.6	48.5	36.0	24.1	17.0
5 NAPTHOCYCLOPARAFFINS	7.9	4.1	13.2	17.9	28.9	39.7	40.5	29.4	15.4
6 FLOURENES	5.9	4.3	4.7	10.4	14.0	17.0	15.9	11.5	7.8
7 PHENANTHRENES	3.0	2.7	3.3	2.5	20.1	14.5	7.2	4.3	3.5
8									

0 TYPE OF RING	10 C19	11 C20	12 C21	13 C22	14
1 ALKYLBENZENES	21.1	9.9	2.9	0.6	
2 BENZOCYCLOPARAFFINS	23.3	11.4	4.1	0.9	
3 BENZODICYCLOPARAFFINS	19.4	9.1	3.1	0.8	
4 NAPTHALENES	10.2	4.7	2.0	0.5	
5 NAPTHOCYCLOPARAFFINS	7.4	3.8	2.3	1.1	
6 FLOURENES	5.0	3.4	2.2	0.9	
7 PHENANTHRENES	3.0	1.6	0.8	0.3	
8					

Table 6. GC-MS data for fraction 3 from the middle distillate cuts of selected EX-6 produced oils.

MIDDISTDI3 8R x 12C

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880331

0 TYPE OF RING	1 C10	2 C11	3 C12	4 C13	5 C14	6 C15	7 C16	8 C17	9 C18
1 ALKYLBENZENES	17.3	21.5	38.1	49.9	51.0	44.4	37.2	25.0	14.8
2 BENZOCYCLOPARAFFINS	16.7	23.3	39.7	58.8	67.7	54.4	40.9	27.9	18.1
3 BENZODICYCLOPARAFFINS	19.2	23.2	17.4	23.1	32.5	39.4	35.5	27.6	19.2
4 NAPTHALENES	32.4	41.6	83.3	100.0	66.5	43.4	23.8	14.5	9.4
5 NAPTHCYCLOPARAFFINS		1.8	6.9	12.7	23.1	28.9	24.1	13.4	6.0
6 FLOURENES		1.3	1.6	7.9	12.3	12.6	8.1	4.8	2.9
7 PHENANTHRENES		0.9	1.1	1.4	15.8	9.1	3.4	2.5	1.7
8									

0 TYPE OF RING	10 C19	11 C20	12 C21
1 ALKYLBENZENES	6.0	1.7	0.1
2 BENZOCYCLOPARAFFINS	9.1	3.0	0.4
3 BENZODICYCLOPARAFFINS	9.4	3.2	0.5
4 NAPTHALENES	5.1	1.8	0.4
5 NAPTHCYCLOPARAFFINS	2.7	1.1	0.3
6 FLOURENES	1.5	0.9	0.3
7 PHENANTHRENES	1.0	0.3	0.0
8			

Table 6. (Cont.)

880608

0 TYPE OF RING	1 C10	2 C11	3 C12	4 C13	5 C14	6 C15	7 C16	8 C17	9 C18
1 ALKYLBENZENES	20.2	26.6	42.4	51.9	48.9	41.7	31.3	18.8	9.7
2 BENZOCYCLOPARAFFINS	19.0	31.8	52.5	75.6	76.1	54.9	38.5	23.8	13.7
3 BENZODICYCLOPARAFFINS	20.1	25.9	20.7	28.9	36.6	41.6	36.7	25.9	15.8
4 NAPTHALENES	41.2	46.4	82.9	100.0	65.8	38.6	21.3	11.6	6.8
5 NAPTHOCYCLOPARAFFINS		1.6	6.9	11.8	21.4	25.3	18.9	9.8	4.2
6 FLOURENES		0.9	1.3	6.6	10.9	10.1	6.0	3.5	1.9
7 PHENANTHRENES		0.6	0.9	1.1	11.6	6.0	2.4	1.6	0.8
8									

0 TYPE OF RING	10 C19	11 C20
1 ALKYLBENZENES	3.0	0.4
2 BENZOCYCLOPARAFFINS	5.5	1.1
3 BENZODICYCLOPARAFFINS	6.2	1.4
4 NAPTHALENES	2.9	0.6
5 NAPTHOCYCLOPARAFFINS	1.4	0.4
6 FLOURENES	0.8	0.4
7 PHENANTHRENES	0.3	
8		

Table 6. (Cont.)

880701

0 TYPE OF RING	1 C10	2 C11	3 C12	4 C13	5 C14	6 C15	7 C16	8 C17	9 C18
1 ALKYLBENZENES	26.1	33.3	46.0	55.9	55.6	49.8	41.1	27.0	15.2
2 BENZOCYCLOPARAFFINS	22.8	36.4	52.6	74.4	78.9	60.5	44.4	30.7	20.8
3 BENZODICYCLOPARAFFINS	21.2	26.9	20.9	28.2	36.6	41.7	38.0	29.2	19.2
4 NAPTHALENES	44.7	50.3	84.0	100.0	64.9	38.6	21.5	12.5	7.3
5 NAPHTHOCYCLOPARAFFINS		2.2	7.7	12.9	22.7	27.7	20.7	9.5	4.0
6 FLOURENES		1.5	1.8	7.8	13.0	11.9	6.0	4.0	3.0
7 PHENANTHRENES		1.0	1.3	1.4	15.6	5.4	2.6	1.8	1.1
8									

0 TYPE OF RING	10 C19	11 C20	12 C21
1 ALKYLBENZENES	6.6	1.8	0.3
2 BENZOCYCLOPARAFFINS	9.4	2.8	0.2
3 BENZODICYCLOPARAFFINS	8.2	2.2	0.0
4 NAPTHALENES	3.3	0.8	0.1
5 NAPHTHOCYCLOPARAFFINS	1.8	0.6	0.2
6 FLOURENES	1.5	0.8	
7 PHENANTHRENES	0.4	0.1	
8			

Table 6. (Cont.)

871102

0 # RINGS	1 C18	2 C19	3 C20	4 C21	5 C22	6 C23	7 C24	8 C25	9 C26	10 C27
1 OPEN CHAIN	9	22	100	245	405	507	480	376	247	139
2 1 RING	17	18	178	543	926	1009	890	646	400	217
3 2 RING	22	36	476	1066	1444	1485	1221	843	512	283
4 3 RING	14	39	626	1172	1404	1508	1171	785	472	275
5 4 RING	0	15	451	1070	1208	1092	816	544	451	252
6 5 RING	0	0	179	386	501	483	414	293	191	123
7 6 RING	0	0	67	131	186	195	183	139	93	58
8										
9										

0 # RINGS	11 C28	12 C29	13 C30	14 C31
1 OPEN CHAIN	71	30	11	0
2 1 RING	113	56	23	7
3 2 RING	138	73	31	9
4 3 RING	145	75	29	8
5 4 RING	126	68	23	6
6 5 RING	57	30	10	0
7 6 RING	27	12	0	0
8				
9				

Table 7. GC-MS data for fraction 1 from the gas oil distillation cuts of selected EX-6 produced oils.

880331

0 # RINGS	1 C18	2 C19	3 C20	4 C21	5 C22	6 C23	7 C24	8 C25	9 C26	10 C27
1 OPEN CHAIN	6	17	104	263	477	577	524	439	355	257
2 1 RING	12	20	223	627	990	853	943	792	617	450
3 2 RING	14	43	521	1078	1397	1472	1325	1088	843	629
4 3 RING	11	53	615	1069	1277	1474	1278	1042	836	672
5 4 RING	0	23	409	886	1001	1043	893	715	640	672
6 5 RING	0	8	156	288	361	397	372	323	295	278
7 6 RING	0	0	46	88	108	138	140	125	112	100
8										

0 # RINGS	11 C28	12 C29	13 C30	14 C31	15 C32
1 OPEN CHAIN	187	127	77	38	40
2 1 RING	323	210	124	61	24
3 2 RING	442	289	163	78	31
4 3 RING	472	321	168	71	26
5 4 RING	421	297	136	51	16
6 5 RING	175	118	59	24	7
7 6 RING	65	45	24	9	0
8					

Table 7. (Cont.)

880608

0 # RINGS	1 C18	2 C19	3 C20	4 C21	5 C22	6 C23	7 C24	8 C25	9 C26	10 C27
1 OPEN CHAIN	12	53	164	285	379	396	358	288	206	136
2 1 RING	85	206	538	883	1016	947	798	612	440	287
3 2 RING	151	367	973	1427	1493	1398	1157	874	620	401
4 3 RING	142	379	1000	1342	1367	1396	1136	850	597	402
5 4 RING	75	181	640	1059	1069	970	770	555	414	342
6 5 RING	23	63	237	346	388	366	319	245	184	133
7 6 RING	0	12	61	96	116	135	108	86	64	44
8										
9										

0 # RINGS	11 C28	12 C29	13 C30	14 C31
1 OPEN CHAIN	85	48	20	8
2 1 RING	176	93	40	11
3 2 RING	235	123	51	14
4 3 RING	226	117	45	12
5 4 RING	167	85	28	5
6 5 RING	66	32	9	0
7 6 RING	20	7	0	0
8				
9				

Table 7. (Cont.)

880701

0 # RINGS	1 C18	2 C19	3 C20	4 C21	5 C22	6 C23	7 C24	8 C25	9 C26	10 C27
1 OPEN CHAIN	0	26	144	296	406	410	342	265	185	121
2 1 RING	16	73	453	876	1018	908	736	552	396	272
3 2 RING	28	170	937	1468	1498	1330	1046	792	561	383
4 3 RING	26	206	1049	1430	1389	1336	1046	768	549	398
5 4 RING	11	92	695	1155	1099	944	716	510	395	366
6 5 RING	0	32	273	405	419	366	297	222	166	133
7 6 RING	0	6	74	119	128	114	100	72	51	35
8										

0 # RINGS	11 C28	12 C29	13 C30	14 C31	15 C32
1 OPEN CHAIN	79	50	25	15	0
2 1 RING	182	108	55	20	0
3 2 RING	248	145	72	24	5
4 3 RING	249	151	66	21	0
5 4 RING	202	125	45	10	0
6 5 RING	69	38	22	0	0
7 6 RING	16	7	0	0	0
8					

Table 7. (Cont.)

871102

0 TYPE OF RING	1 C18	2 C19	3 C20	4 C21	5 C22	6 C23	7 C24	8 C25	9 C26
1 ALKYLBENZENES	5.1	6.4	22.8	47.5	59.0	55.3	45.7	32.9	21.9
2 BENZOCYCLOPARAFFINS	9.6	18.1	49.9	84.2	100.0	91.4	77.1	55.6	37.5
3 BENZODICYCLOPARAFFINS	10.3	17.7	37.6	60.2	74.9	72.3	64.2	49.2	36.2
4 NAPTHALENES	8.1	17.5	23.0	31.9	41.0	40.8	38.2	31.0	24.2
5 NAPTHOCYCLOPARAFFINS	4.7	6.8	6.5	9.4	15.8	24.3	25.4	22.6	18.6
6 FLOURENES		2.3	2.3	4.0	8.6	14.6	15.8	14.8	12.4
7 PHENANTHRENES		1.5	1.6	3.2	6.6	10.4	10.7	9.7	7.6
8									

0 TYPE OF RING	10 C27	11 C28	12 C29	13 C30	14 C31	15 C32	16 C33
1 ALKYLBENZENES	14.3	9.2	5.6	2.9	1.1	0.3	0.1
2 BENZOCYCLOPARAFFINS	24.9	15.7	9.2	4.6	1.9	0.6	0.1
3 BENZODICYCLOPARAFFINS	26.0	17.7	10.9	5.3	2.1	0.7	0.2
4 NAPTHALENES	18.6	12.7	8.2	4.2	1.7	0.6	0.2
5 NAPTHOCYCLOPARAFFINS	14.6	9.9	6.3	3.5	1.7	0.7	0.2
6 FLOURENES	9.7	6.7	4.4	2.6	1.4	0.6	0.2
7 PHENANTHRENES	5.9	4.2	2.9	1.8	0.9	0.4	0.1
8							

Table 8. GC-MS data for fraction 2 from the gas oil distillation cuts of selected EX-6 produced oils.

0 TYPE OF RING	1 C18	2 C19	3 C20	4 C21	5 C22	6 C23	7 C24	8 C25	9 C26
1 ALKYLBENZENES	10.6	10.2	16.8	33.5	46.7	46.5	40.0	31.4	23.8
2 BENZOCYCLOPARAFFINS	14.6	15.8	28.7	47.1	62.6	62.8	57.3	46.7	36.2
3 BENZODICYCLOPARAFFINS	14.0	15.5	24.0	37.6	45.9	47.4	45.3	39.0	32.5
4 NAPTHALENES	11.9	27.2	22.4	21.7	26.2	26.5	26.9	24.1	21.2
5 NAPTHOCYCLOPARAFFINS	7.7	12.3	8.8	7.8	10.2	15.0	17.0	16.3	14.9
6 FLOURENES	5.9	4.5	3.5	3.9	6.7	11.1	12.8	12.1	10.6
7 PHENANTHRENES		3.6	3.2	3.8	6.2	9.7	10.7	9.8	7.9
8									

0 TYPE OF RING	10 C27	11 C28	12 C29	13 C30	14 C31	15 C32	16 C33
1 ALKYLBENZENES	18.1	11.9	7.4	3.9	1.6	0.5	0.1
2 BENZOCYCLOPARAFFINS	26.8	17.8	11.2	6.1	2.7	0.8	0.2
3 BENZODICYCLOPARAFFINS	25.5	18.4	11.8	6.4	2.8	1.0	0.2
4 NAPTHALENES	18.2	13.2	9.1	5.0	2.2	0.7	0.2
5 NAPTHOCYCLOPARAFFINS	13.0	9.9	6.9	3.9	1.9	0.8	0.2
6 FLOURENES	9.2	7.1	5.0	3.0	1.5	0.7	0.2
7 PHENANTHRENES	6.7	5.2	3.7	2.3	1.2	0.5	0.1
8							

Table 8. (Cont.)

880608

0 TYPE OF RING	1 C18	2 C19	3 C20	4 C21	5 C22	6 C23	7 C24	8 C25	9 C26
1 ALKYLBENZENES	22.3	35.8	47.9	56.0	50.9	42.4	34.1	26.3	19.3
2 BENZOCYCLOPARAFFINS	46.3	71.6	91.8	100.0	91.1	73.4	60.6	46.5	34.2
3 BENZODICYCLOPARAFFINS	45.9	70.0	78.6	83.6	79.2	67.5	57.8	45.7	35.6
4 NAPTHALENES	25.0	49.2	55.5	52.0	50.6	43.2	37.9	31.0	25.0
5 NAPHTHOCYCLOPARAFFINS	11.0	15.7	15.7	17.9	21.9	23.6	22.5	19.6	16.6
6 FLOURENES	6.7	6.4	7.5	9.9	12.6	14.2	13.3	11.9	10.3
7 PHENANTHRENES		5.2	6.3	8.7	9.2	9.9	9.5	8.2	6.8
8									

0 TYPE OF RING	10 C27	11 C28	12 C29	13 C30	14 C31	15 C32	16 C33	17
1 ALKYLBENZENES	15.0	9.4	5.6	2.7	1.0	0.3	0.1	
2 BENZOCYCLOPARAFFINS	24.4	15.0	8.5	4.2	1.7	0.1	0.1	
3 BENZODICYCLOPARAFFINS	26.7	17.5	10.0	4.6	1.8	0.5	0.1	
4 NAPTHALENES	19.5	12.6	7.7	3.7	1.5	0.5	0.1	
5 NAPHTHOCYCLOPARAFFINS	13.1	8.9	5.7	3.1	1.4	0.5	0.2	
6 FLOURENES	8.6	6.3	4.2	2.5	1.3	0.5	0.2	
7 PHENANTHRENES	5.6	4.3	3.0	1.9	0.9	0.4	0.1	
8								

Table 8. (Cont.)

880701

0 TYPE OF RING	1 C18	2 C19	3 C20	4 C21	5 C22	6 C23	7 C24	8 C25	9 C26
1 ALKYLBENZENES	9.4	23.7	42.5	57.3	57.8	51.6	45.9	38.4	31.7
2 BENZOCYCLOPARAFFINS	21.8	53.6	82.9	99.9	100.0	87.0	77.2	64.4	53.7
3 BENZODICYCLOPARAFFINS	25.6	55.6	73.3	85.7	85.9	78.3	73.1	63.4	55.2
4 NAPTHALENES	17.7	42.0	53.5	54.8	56.0	51.5	49.0	43.3	39.1
5 NAPTHOCYCLOPARAFFINS	7.5	12.4	14.4	20.5	26.0	30.3	30.8	28.6	26.5
6 FLOURENES	4.2	4.0	5.6	9.3	14.1	18.1	18.7	18.0	16.7
7 PHENANTHRENES		3.1	4.8	7.8	9.5	11.7	12.0	11.4	10.4
8									

0 TYPE OF RING	10 C27	11 C28	12 C29	13 C30	14 C31	15 C32	16 C33	17 C34
1 ALKYLBENZENES	26.5	20.0	14.2	8.9	4.9	2.3	1.0	0.3
2 BENZOCYCLOPARAFFINS	43.1	31.0	21.5	13.2	7.1	3.3	1.5	0.5
3 BENZODICYCLOPARAFFINS	46.8	35.7	24.7	14.4	7.3	3.5	1.6	0.6
4 NAPTHALENES	35.2	26.6	19.5	11.3	5.8	2.8	1.3	0.5
5 NAPTHOCYCLOPARAFFINS	23.7	18.2	13.7	8.8	5.2	2.9	1.4	0.6
6 FLOURENES	15.2	12.8	9.8	6.9	4.4	2.6	1.3	0.6
7 PHENANTHRENES	9.6	8.3	6.7	5.0	3.3	1.9	1.0	0.4
8								

0 TYPE OF RING	18 C35
1 ALKYLBENZENES	0.1
2 BENZOCYCLOPARAFFINS	0.1
3 BENZODICYCLOPARAFFINS	0.2
4 NAPTHALENES	0.1
5 NAPTHOCYCLOPARAFFINS	0.2
6 FLOURENES	0.2
7 PHENANTHRENES	0.1

Table 8. (Cont.)

871102

0 TYPE OF RING	1 C14	2 C15	3 C16	4 C17	5 C18	6 C19	7 C20	8 C21	9 C22
1 ALKYLBENZENES	9.7	8.7	12.7	18.3	22.3	29.2	31.3	25.0	17.9
2 BENZOCYCLOPARAFFINS		9.3	6.7	10.3	18.4	24.5	29.0	25.2	19.0
3 BENZODICYCLOPARAFFINS		5.4	7.8	11.0	12.7	18.5	21.8	20.6	16.1
4 DIBENZOTHIOPHENES		13.8	61.0	100.0	72.5	53.0	38.8	28.6	19.9
5 NAPTHOCYCLOPARAFFINS		9.6	13.5	28.6	46.8	43.8	34.1	26.1	17.9
6 FLOURENES		11.0	19.0	31.0	36.3	35.5	27.8	20.6	13.8
7 PHENANTHRENES		17.3	62.8	55.5	31.2	18.8	14.6	11.8	8.5
8									

0 TYPE OF RING	10 C23	11 C24	12 C25	13 C26
1 ALKYLBENZENES	9.8	4.5	1.5	0.2
2 BENZOCYCLOPARAFFINS	10.4	4.9	1.6	0.2
3 BENZODICYCLOPARAFFINS	10.1	5.2	1.8	0.3
4 DIBENZOTHIOPHENES	11.5	5.8	2.1	0.5
5 NAPTHOCYCLOPARAFFINS	10.8	5.8	2.3	0.6
6 FLOURENES	8.1	4.2	1.8	0.4
7 PHENANTHRENES	5.8	3.3	1.4	0.3
8				

Table 9. GC-MS data for fraction 3 from the gas oil distillation cuts of selected EX-6 produced oils.

880331

0 TYPE OF RING	1 C14	2 C15	3 C16	4 C17	5 C18	6 C19	7 C20	8 C21	9 C22
1 ALKYLBENZENES	12.9	10.6	14.7	23.2	30.9	39.6	41.1	34.3	25.9
2 BENZOCYCLOPARAFFINS	10.5	9.2	9.6	14.7	23.6	28.9	34.3	32.8	28.5
3 BENEZODICYCLOPARAFFINS		8.1	10.9	13.1	15.0	21.5	26.3	26.9	24.5
4 DIBENZOTHIOPHENES		17.2	70.1	100.0	75.5	61.9	49.4	39.8	30.5
5 NAPHTHOCYCLOPARAFFINS		11.1	17.7	32.6	50.6	48.8	41.0	34.3	26.1
6 FLOURENES		12.3	22.2	33.5	38.7	39.2	34.1	27.9	22.5
7 PHENANTHRENES		19.4	56.4	53.0	34.3	23.7	20.9	18.7	15.1
8									

0 TYPE OF RING	10 C23	11 C24	12 C25	13 C26	14 C27	15 C28	16 C29
1 ALKYLBENZENES	17.8	11.6	7.0	3.9	1.9	0.7	0.2
2 BENZOCYCLOPARAFFINS	20.3	13.7	8.3	4.7	2.3	0.9	0.2
3 BENEZODICYCLOPARAFFINS	18.6	13.3	8.3	4.9	2.7	1.2	0.4
4 DIBENZOTHIOPHENES	21.1	13.7	8.0	4.6	2.7	1.2	0.4
5 NAPHTHOCYCLOPARAFFINS	18.9	12.9	7.9	4.7	2.5	1.1	0.4
6 FLOURENES	15.5	10.3	6.3	3.8	1.9	0.8	0.3
7 PHENANTHRENES	11.6	7.9	4.9	2.9	1.6	0.7	0.2
8							

Table 9. (Cont.)

880608

0 TYPE OF RING	1 C14	2 C15	3 C16	4 C17	5 C18	6 C19	7 C20	8 C21	9 C22
1 ALKYLBENZENES	12.9	11.2	17.0	27.8	36.8	42.5	39.0	29.9	21.1
2 BENZOCYCLOPARAFFINS	9.6	8.7	11.1	19.3	26.8	30.7	31.1	25.0	19.5
3 BENZODICYCLOPARAFFINS		7.2	11.0	15.4	18.9	23.1	22.7	19.0	15.0
4 DIBENZOTHIOPHENES		24.2	74.6	100.0	82.0	65.0	47.3	32.8	21.8
5 NAPHTHOCYCLOPARAFFINS		15.9	27.3	44.7	57.0	51.7	41.2	31.4	22.0
6 FLOURENES		16.6	27.9	37.0	40.6	40.4	35.4	27.8	20.3
7 PHENANTHRENES		27.6	61.1	54.5	36.1	25.0	21.0	17.3	12.9
8									

0 TYPE OF RING	10 C23	11 C24	12 C25	13 C26	14 C27	15 C28
1 ALKYLBENZENES	13.2	7.8	4.2	1.7	0.7	0.1
2 BENZOCYCLOPARAFFINS	12.5	7.4	3.8	2.0	0.6	0.1
3 BENZODICYCLOPARAFFINS	10.1	6.3	3.2	1.5	0.5	0.1
4 DIBENZOTHIOPHENES	12.9	7.1	3.4	1.5	0.6	0.1
5 NAPHTHOCYCLOPARAFFINS	13.7	8.3	4.4	2.1	0.8	0.2
6 FLOURENES	12.3	7.3	3.9	1.9	0.8	0.2
7 PHENANTHRENES	9.0	5.6	3.2	1.7	0.7	0.2
8						

Table 9. (Cont.)

880701

0 TYPE OF RING	1 C14	2 C15	3 C16	4 C17	5 C18	6 C19	7 C20	8 C21	9 C22
1 ALKYLBENZENES	11.6	9.8	14.4	36.6	43.0	39.2	28.7	20.2	12.8
2 BENZOCYCLOPARAFFINS		7.0	8.9	26.2	29.4	29.0	22.1	17.3	11.4
3 BENZODICYCLOPARAFFINS		5.6	9.4	16.3	20.5	19.1	15.7	12.2	8.2
4 DIBENZOTHIOPHENES		20.2	76.7	80.8	64.1	46.1	31.2	20.7	12.4
5 NAPHTHOCYCLOPARAFFINS		13.4	25.0	60.6	52.3	41.1	31.5	21.5	13.7
6 FLOURENES		14.6	29.8	41.4	40.5	35.1	28.1	20.7	12.8
7 PHENANTHRENES		30.7	69.4	36.7	25.3	21.6	17.9	13.3	9.3
8									

0 TYPE OF RING	10 C23	11 C24	12 C25	13 C26	14 C27
1 ALKYLBENZENES	7.8	4.5	2.3	0.9	0.2
2 BENZOCYCLOPARAFFINS	7.0	3.9	1.9	0.7	0.1
3 BENZODICYCLOPARAFFINS	5.2	2.7	1.2	0.4	0.1
4 DIBENZOTHIOPHENES	6.8	3.1	1.4	0.5	0.1
5 NAPHTHOCYCLOPARAFFINS	6.3	4.4	2.2	0.8	0.2
6 FLOURENES	7.7	4.1	2.0	0.8	0.2
7 PHENANTHRENES	5.9	3.3	1.7	0.7	0.2
8					

Table 9. (Cont.)

RING9 9R x 16C

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871102

0 # RINGS	1 C26	2 C27	3 C28	4 C29	5 C30	6 C31	7 C32	8 C33	9 C34	10 C35
1 OPEN CHAIN	20.31	18.39	17.87	16.54	15.56	15.02	15.80	15.16	14.76	12.63
2 1 RING	39.39	34.11	28.49	24.91	23.70	24.29	24.28	23.91	22.34	19.16
3 2 RING	67.52	59.52	53.17	50.14	50.62	50.92	50.11	59.08	44.31	38.26
4 3 RING	72.86	71.10	72.62	93.55	100.76	90.62	82.38	80.36	71.75	61.93
5 4 RING	67.54	90.51	115.30	182.00	169.31	126.79	109.65	100.71	85.47	71.85
6 5 RING	40.13	69.68	65.68	100.92	106.94	99.51	83.51	74.94	63.37	54.78
7 6 RING	10.69	15.41	15.41	20.94	23.60	25.17	24.25	24.10	21.85	20.16
8										
9										

0 # RINGS	11 C36	12 C37	13 C38	14 C39	15 C40	16 C41
1 OPEN CHAIN	11.09	9.05	8.61	8.82	4.90	3.32
2 1 RING	15.76	12.65	10.86	10.09	6.81	4.59
3 2 RING	32.01	15.73	22.19	19.17	16.12	11.03
4 3 RING	49.13	39.83	35.42	30.91	26.70	18.49
5 4 RING	54.55	44.63	39.45	33.92	30.04	21.31
6 5 RING	41.19	37.04	29.91	25.99	24.33	16.54
7 6 RING	17.70	15.65	13.73	12.75	13.73	8.13
8						
9						

Table 10. GC-MS data for fraction 1 from the residue distillation cuts of selected EX-6 produced oils.

880331

0 # RINGS	1 C26	2 C27	3 C28	4 C29	5 C30	6 C31	7 C32	8 C33	9 C34	10 C35
1 OPEN CHAIN	19.93	18.63	18.33	18.43	19.05	19.30	20.67	20.79	20.61	19.16
2 1 RING	33.89	29.53	25.98	24.28	24.67	26.23	27.02	27.21	26.29	23.42
3 2 RING	52.78	47.31	43.29	42.91	45.40	45.89	45.98	46.18	43.58	38.83
4 3 RING	55.53	55.41	61.00	80.53	83.85	73.84	68.89	69.33	63.83	56.83
5 4 RING	55.04	84.61	106.13	162.50	156.02	118.52	103.42	92.54	81.08	71.70
6 5 RING	40.42	85.99	78.01	126.15	138.83	122.28	94.91	83.16	72.28	65.16
7 6 RING	12.01	21.25	16.32	22.66	25.65	29.63	28.27	27.37	25.97	25.24
8										

0 # RINGS	11 C36	12 C37	13 C38	14 C39	15 C40	16 C41	17 C42	18 C43	19 C44
1 OPEN CHAIN	17.01	14.60	13.74	13.78	8.72	6.12	4.35	2.98	1.79
2 1 RING	20.73	17.72	16.01	14.97	10.59	7.49	5.32	3.58	2.26
3 2 RING	33.95	28.35	25.60	22.67	18.48	13.11	9.64	6.94	4.78
4 3 RING	46.90	39.52	36.00	31.62	27.00	18.81	14.05	10.39	7.36
5 4 RING	54.46	44.59	39.55	33.60	29.04	20.13	15.01	11.01	7.78
6 5 RING	45.34	40.25	32.39	27.56	24.49	16.13	11.70	8.30	5.62
7 6 RING	22.27	20.66	18.07	15.95	15.87	9.40	6.63	4.48	2.96
8									

Table 10. (Cont.)

880331

0 # RINGS	20 C45	21 C46	22 C47	23 C48	24 C49
1 OPEN CHAIN	1.07	0.34	0.08	0.00	0.00
2 1 RING	1.32	0.71	0.29	0.05	0.06
3 2 RING	3.03	1.51	0.72	0.24	0.06
4 3 RING	4.59	2.40	1.20	0.45	0.08
5 4 RING	4.96	2.49	1.15	0.35	0.03
6 5 RING	3.45	1.75	0.64	0.14	0.00
7 6 RING	1.77	0.84	0.27	0.04	0.00
8					

Table 10. (Cont.)

880608

0 # RINGS	1 C26	2 C27	3 C28	4 C29	5 C30	6 C31	7 C32	8 C33	9 C34	10 C35
1 OPEN CHAIN	17.92	16.65	17.35	17.14	17.67	18.15	18.09	16.99	15.88	13.82
2 1 RING	29.01	25.24	23.50	24.10	26.56	27.11	24.53	22.94	20.64	17.63
3 2 RING	45.79	42.51	42.80	46.73	47.77	46.17	43.08	40.55	35.85	30.39
4 3 RING	52.83	59.76	72.10	96.71	96.25	76.92	66.85	62.29	53.26	45.58
5 4 RING	59.81	102.23	129.95	181.46	161.32	104.97	91.26	80.35	67.42	57.53
6 5 RING	39.93	83.29	76.79	120.41	126.20	108.69	82.27	69.45	58.43	51.67
7 6 RING	12.01	20.83	17.67	24.70	26.31	27.86	24.76	23.38	21.09	19.70
8										
9										

0 # RINGS	11 C36	12 C37	13 C38	14 C39	15 C40	16 C41	17 C42	18 C43	19 C44
1 OPEN CHAIN	11.73	10.01	9.23	9.28	5.42	3.57	2.39	1.50	0.86
2 1 RING	14.76	12.09	10.87	10.06	6.67	4.54	3.14	1.99	1.19
3 2 RING	25.29	20.50	18.22	15.74	12.50	8.59	6.06	4.20	2.70
4 3 RING	36.39	29.48	26.34	22.78	18.96	12.84	8.20	6.58	4.52
5 4 RING	42.14	34.00	29.39	24.89	20.91	13.95	10.04	7.17	4.87
6 5 RING	35.05	30.61	24.06	20.14	17.63	11.23	7.77	5.32	3.44
7 6 RING	16.96	15.25	13.09	11.47	11.26	6.34	4.33	2.79	1.74
8									
9									

Table 10. (Cont.)

880608

0 # RINGS	20 C45	21 C46	22 C47	23 C48
1 OPEN CHAIN	0.46	0.11	0.01	0.01
2 1 RING	0.69	0.30	0.08	0.08
3 2 RING	1.61	0.68	0.34	0.15
4 3 RING	2.66	1.35	0.55	0.12
5 4 RING	2.94	1.34	0.57	0.04
6 5 RING	1.98	0.92	0.30	0.00
7 6 RING	0.94	0.38	0.07	0.00
8				
9				

Table 10. (Cont.)

880701

0 # RINGS	1 C26	2 C27	3 C28	4 C29	5 C30	6 C31	7 C32	8 C33	9 C34	10 C35
1 OPEN CHAIN	20.37	18.79	18.87	19.67	20.89	21.13	20.91	19.62	18.16	15.26
2 1 RING	34.05	29.38	25.79	25.09	27.47	28.04	27.21	25.89	22.98	19.36
3 2 RING	53.59	49.03	45.41	46.75	50.64	48.94	47.48	44.59	39.09	23.11
4 3 RING	57.11	59.72	67.41	90.04	92.59	78.01	70.05	65.49	56.23	48.39
5 4 RING	58.45	94.17	119.80	176.67	163.30	122.54	98.97	84.02	69.80	60.08
6 5 RING	41.52	88.76	82.08	128.73	137.97	117.07	89.62	75.06	61.51	53.31
7 6 RING	11.76	19.43	16.57	22.81	26.15	28.68	26.18	25.10	22.30	20.77
8										

0 # RINGS	11 C36	12 C37	13 C38	14 C39	15 C40	16 C41	17 C42	18 C43	19 C44
1 OPEN CHAIN	12.96	10.59	9.66	9.48	5.76	3.88	2.65	1.68	1.04
2 1 RING	16.10	12.96	11.54	10.58	7.12	4.95	3.50	2.41	1.52
3 2 RING	26.83	22.09	19.04	16.61	13.34	9.25	6.70	4.81	3.26
4 3 RING	38.00	30.62	27.41	23.80	19.73	13.63	9.97	7.46	5.30
5 4 RING	43.53	34.75	30.24	25.36	26.62	14.75	10.95	8.02	5.71
6 5 RING	36.53	31.59	24.83	20.78	18.22	11.95	8.49	5.98	4.05
7 6 RING	17.77	15.70	13.44	11.70	11.38	6.54	5.50	2.99	1.90
8									

Table 10. (Cont.)

880701

0 # RINGS	20 C45	21 C46	22 C47	23 C48	24 C49
1 OPEN CHAIN	0.53	0.16	0.02	0.00	0.00
2 1 RING	0.83	0.32	0.07	0.00	0.00
3 2 RING	2.03	0.95	0.38	0.09	0.02
4 3 RING	3.40	1.66	0.79	0.25	0.03
5 4 RING	3.59	1.87	0.91	0.27	0.00
6 5 RING	2.48	1.23	0.44	0.12	0.00
7 6 RING	1.08	0.45	0.12	0.01	0.00
8					

Table 10. (Cont.)

871102

0 TYPE OF RING	1 C26	2 C27	3 C28	4 C29	5 C30	6 C31	7 C32	8 C33	9 C34
1 ALKYLBENZENES	0.1	4.4	16.0	32.9	37.9	24.6	21.5	17.4	13.4
2 BENZOCYCLOPARAFFINS	0.4	32.7	38.7	87.5	100.0	73.3	50.7	38.5	27.1
3 BENZODICYCLOPARAFFINS	0.3	5.0	12.2	33.2	48.9	51.1	49.9	43.6	34.0
4 NAPTHALENES	10.4	25.5	19.3	41.7	53.3	51.5	48.7	45.6	35.2
5 NAPTHOCYCLOPARAFFINS	2.9	8.7	13.9	30.9	37.5	34.3	33.9	30.8	23.1
6 FLOURENES		0.4	2.7	8.8	13.2	15.3	15.6	15.0	15.8
7 PHENANTHRENES			0.1	1.5	4.7	6.4	7.5	7.3	7.6
8									4.0
9									

0 TYPE OF RING	10 C35	11 C36	12 C37	13 C38
1 ALKYLBENZENES	9.0	4.2	0.8	0.1
2 BENZOCYCLOPARAFFINS	17.5	10.2	3.0	1.2
3 BENZODICYCLOPARAFFINS	22.6	13.3	7.2	2.0
4 NAPTHALENES	23.1	13.7	7.2	2.1
5 NAPTHOCYCLOPARAFFINS	15.8	8.7	4.2	0.9
6 FLOURENES	7.6	4.3	6.9	0.2
7 PHENANTHRENES	4.0	1.6	0.2	
8				
9				

Table 11. GC-MS data for fraction 2 from the residue distillation cuts of selected EX-6 produced oils.

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880331

0 TYPE OF RING	1 C28	2 C29	3 C30	4 C31	5 C32	6 C33	7 C34	8 C35	9 C36
1 ALKYLBENZENES	2.7	11.9	18.8	16.5	20.3	25.5	28.9	32.4	30.4
2 BENZOCYCLOPARAFFINS	20.5	53.6	76.5	62.7	58.5	62.1	65.0	68.2	57.1
3 BENZODICYCLOPARAFFINS	5.2	15.5	29.9	46.4	62.9	77.3	85.1	87.7	80.0
4 NAPTHALENES	8.2	21.8	35.4	52.8	73.5	90.7	96.7	100.0	91.1
5 NAPHTHOCYCLOPARAFFINS	8.0	18.7	33.2	46.0	62.9	75.5	82.2	82.8	78.1
6 FLOURENES	1.7	6.0	13.7	24.5	34.4	43.2	48.7	51.6	51.1
7 PHENANTHRENES	.	0.3	3.0	7.6	12.8	18.7	21.6	24.8	26.4
8									

0 TYPE OF RING	10 C37	11 C38	12 C39	13 C40	14 C41	15 C42	16 C43	17 C44
1 ALKYLBENZENES	26.6	24.9	26.1	16.9	12.9	9.7	5.3	2.3
2 BENZOCYCLOPARAFFINS	55.0	43.2	38.3	33.2	22.6	17.9	11.5	7.3
3 BENZODICYCLOPARAFFINS	70.9	64.1	57.9	59.2	37.1	28.2	20.1	13.5
4 NAPTHALENES	82.7	73.0	67.6	68.0	43.3	32.8	24.1	16.5
5 NAPHTHOCYCLOPARAFFINS	70.7	64.7	61.2	66.3	38.4	28.7	20.5	14.0
6 FLOURENES	48.9	43.9	44.1	51.3	28.1	20.7	14.4	9.2
7 PHENANTHRENES	26.0	24.2	25.4	30.4	16.5	11.7	9.8	4.6
8	DEL							

Table 11. (Cont.)

880331

0 TYPE OF RING	18 C45	19 C46	20 C47	21 C48	22
1 ALKYLBENZENES	1.0	0.1	0.0	0.0	
2 BENZOCYCLOPARAFFINS	3.4	1.3	0.2	0.0	
3 BENZODICYCLOPARAFFINS	7.3	2.4	0.7	0.1	
4 NAPTHALENES	11.2	3.7	1.2	0.3	
5 NAPHTHOCYCLOPARAFFINS	8.4	3.8	1.1	0.2	
6 FLOURENES	5.2	2.2	0.5	0.1	
7 PHENANTHRENES	2.4	0.9	0.0	0.0	
8					

Table 11. (Cont.)

880608

0 TYPE OF RING	1 C28	2 C29	3 C30	4 C31	5 C32	6 C33	7 C34	8 C35	9 C36
1 ALKYLBENZENES	18.0	32.0	40.7	44.7	49.5	49.2	48.5	47.7	44.4
2 BENZOCYCLOPARAFFINS	38.1	66.1	90.3	94.3	92.7	89.2	85.1	82.7	71.1
3 BENZODICYCLOPARAFFINS	36.7	64.2	79.4	93.7	100.0	97.4	94.7	89.0	82.2
4 NAPTHALENES	41.4	71.7	79.3	83.3	87.3	89.8	87.6	83.6	77.3
5 NAPTHOCYCLOPARAFFINS	31.0	47.3	55.0	60.8	65.6	70.2	70.1	68.5	63.5
6 FLOURENES	15.0	21.6	29.2	35.2	39.6	43.1	46.1	46.4	45.4
7 PHENANTHRENES	7.2	10.5	15.3	19.8	23.9	27.6	29.2	30.5	30.8
8									

0 TYPE OF RING	10 C37	11 C38	12 C39	13 C40	14 C41	15 C42	16 C43	17 C44
1 ALKYLBENZENES	41.2	39.6	37.7	30.3	24.5	21.0	16.2	12.4
2 BENZOCYCLOPARAFFINS	70.6	60.1	55.0	46.1	37.6	30.9	23.7	18.6
3 BENZODICYCLOPARAFFINS	77.5	71.4	63.4	60.5	44.4	36.8	28.7	22.2
4 NAPTHALENES	71.9	65.6	61.5	58.7	43.8	36.1	28.6	21.9
5 NAPTHOCYCLOPARAFFINS	59.6	55.7	52.5	53.2	37.7	31.7	25.6	19.3
6 FLOURENES	43.9	41.2	41.1	41.8	29.1	24.2	20.0	15.1
7 PHENANTHRENES	29.7	28.9	30.0	30.4	21.5	18.2	16.0	11.7
8								

Table 11. (Cont.)

880608

0 TYPE OF RING	18 C45	19 C46	20 C47	21 C48	22 C49	23 C50	24 C51
1 ALKYLBENZENES	9.3	6.7	4.4	2.6	1.2	0.4	
2 BENZOCYCLOPARAFFINS	13.5	9.9	6.4	3.3	1.4	0.7	
3 BENZODICYCLOPARAFFINS	16.5	11.3	7.3	4.3	1.7	0.8	
4 NAPTHALENES	16.7	11.6	7.7	4.6	2.1	0.9	0.2
5 NAPTHOCYCLOPARAFFINS	14.5	10.4	7.2	4.8	2.4	0.8	0.2
6 FLOURENES	11.4	8.5	5.8	3.7	1.8	0.7	0.2
7 PHENANTHRENES	9.0	6.6	4.8	3.2	1.8	0.7	0.2
8							

Table 11. (Cont.)

880701

0 type of ring	1 C28	2 C29	3 C30	4 C31	5 C32	6 33	7 C34	8 C35	9 C36
1 alkylbenzenes	8.1	16.1	26.2	37.4	45.2	49.4	51.2	51.3	49.1
2 benzocycloparaffins	17.6	37.3	55.2	73.5	83.5	85.8	85.0	84.0	76.7
3 benzodicycloparaffins	21.4	42.9	84.1	84.1	95.1	100.0	99.8	96.7	90.0
4 napthalenes	29.6	49.8	80.3	80.3	88.9	95.8	95.1	92.8	85.7
5 napthocycloparaffins	24.3	41.2	49.8	58.4	68.9	76.1	76.8	75.4	71.9
6 flourenes	10.7	18.1	25.4	33.3	40.6	46.2	50.3	51.1	50.8
7 phenanthrenes	4.4	7.2	11.1	17.2	23.0	28.0	30.7	32.8	34.4
8									
9									

0 type of ring	10 C37	11 C38	12 C39	13 C40	14 C41	15 C42	16 C43	17 C44
1 alkylbenzenes	46.5	45.4	43.0	35.0	29.5	25.3	19.6	14.9
2 benzocycloparaffins	77.3	67.3	62.3	54.6	43.1	36.6	28.7	22.6
3 benzodicycloparaffins	84.0	77.7	73.1	69.3	52.5	44.3	34.9	27.8
4 napthalenes	80.2	74.7	70.8	67.8	51.1	42.9	34.9	27.4
5 napthocycloparaffins	67.9	64.3	61.7	62.6	45.4	38.9	31.9	27.3
6 flourenes	49.9	48.1	47.9	49.9	35.2	30.0	25.1	19.1
7 phenanthrenes	33.6	33.2	35.3	36.3	26.1	22.3	20.0	14.3
8								
9								

Table 11. (Cont.)

880701

0 type of ring	18 C45	19 C46	20 C47	21 C48	22 C49	23 C50	24
1 alkylbenzenes	11.4	8.0	5.4	3.0	1.6	0.4	
2 benzocycloparaffins	17.0	12.6	8.3	5.2	2.4	1.0	
3 benzodicycloparaffins	20.9	14.9	10.5	6.4	3.2	1.1	
4 napthalenes	20.8	14.8	10.3	6.6	3.2	1.4	0.2
5 napthocycloparaffins	18.2	13.2	9.2	6.0	3.2	1.1	0.3
6 flourenes	14.2	10.5	7.2	4.7	2.3	0.9	0.2
7 phenanthrenes	11.1	8.1	5.6	3.7	1.9	0.7	0.2
8							
9							

Table 11. (Cont.)

871102

0 TYPE OF RING	1 C22	2 C23	3 C24	4 C25	5 C26	6 C27	7 C28	8 C29	9 C30
1 ALKYLBENZENES	9.3	15.1	25.1	39.3	55.9	67.9	72.3	73.9	89.0
2 BENZOCYCLOPARAFFINS	11.1	18.6	25.6	37.3	49.7	58.8	66.4	76.9	87.6
3 BENZODICYCLOPARAFFINS		8.4	13.0	20.1	30.5	42.3	52.9	64.7	76.5
4 NAPTHALENES		5.1	7.1	14.7	29.7	53.5	62.3	76.2	83.5
5 NAPHTHOCYCLOPARAFFINS		5.5	8.3	17.2	31.9	50.1	67.5	85.5	88.9
6 FLOURENES		4.3	7.1	15.2	29.4	56.5	82.3	100.0	86.4
7 PHENANTHRENES		4.1	7.7	17.3	31.4	51.5	67.7	73.8	73.1
8									

0 TYPE OF RING	10 C31	11 C32	12 C33	13 C34	14 C35	15 C36	16 C37	17 C38
1 ALKYLBENZENES	82.3	79.1	72.4	65.6	58.1	50.1	43.1	37.2
2 BENZOCYCLOPARAFFINS	92.4	91.4	84.4	75.9	69.1	59.8	56.9	45.1
3 BENZODICYCLOPARAFFINS	80.7	81.7	78.6	71.7	64.6	56.2	49.5	41.7
4 NAPTHALENES	85.2	82.7	79.6	69.5	63.0	53.4	47.6	39.1
5 NAPHTHOCYCLOPARAFFINS	88.8	88.1	82.9	73.3	64.6	55.5	47.1	39.9
6 FLOURENES	82.2	84.9	82.1	69.7	61.2	51.0	43.6	36.5
7 PHENANTHRENES	74.7	70.8	65.4	56.8	50.1	44.5	38.0	32.2
8								

Table 12. GC-MS data for fraction 3 from the residue distillation cuts of selected EX-6 produced oils.

871102

0 TYPE OF RING	18 C39	19 C40	20 C41	21 C42	22 C43	23 C44	24 C45	25 C46
1 ALKYLBENZENES	31.3	22.0	16.4	11.7	7.5	4.7	2.6	1.0
2 BENZOCYCLOPARAFFINS	38.1	29.1	20.8	15.2	9.9	6.6	3.7	2.0
3 BENZODICYCLOPARAFFINS	35.3	28.1	20.0	14.8	9.8	6.4	3.8	1.8
4 NAPTHALENES	33.2	26.3	18.8	13.7	9.1	5.8	3.3	1.5
5 NAPHTHOCYCLOPARAFFINS	32.8	27.5	18.4	13.7	9.0	5.7	3.1	1.4
6 FLOURENES	31.5	25.9	17.1	12.6	8.3	4.8	2.6	1.2
7 PHENANTHRENES	27.3	23.6	15.5	11.1	7.8	4.5	2.4	1.1
8								

0 TYPE OF RING	26 C47	27
1 ALKYLBENZENES	0.4	
2 BENZOCYCLOPARAFFINS	0.7	
3 BENZODICYCLOPARAFFINS	0.7	
4 NAPTHALENES	0.5	
5 NAPHTHOCYCLOPARAFFINS	0.4	
6 FLOURENES	0.3	
7 PHENANTHRENES	0.3	
8		

Table 12. (Cont.)

880331

0 TYPE OF RING	1 C22	2 C23	3 C24	4 C25	5 C26	6 C27	7 C28	8 C29	9 C30
1 ALKYLBENZENES	11.6	18.7	27.1	43.7	58.5	69.7	75.2	79.0	91.8
2 BENZOCYCLOPARAFFINS	13.5	21.9	28.7	40.6	51.7	61.2	70.2	82.5	92.9
3 BENZODICYCLOPARAFFINS		10.3	16.0	22.9	33.3	45.7	56.9	69.7	79.6
4 NAPTHALENES		6.7	10.2	20.8	38.7	62.7	69.3	83.1	88.3
5 NAPHTHOCYCLOPARAFFINS		7.7	13.4	24.4	40.9	57.0	74.8	90.5	91.6
6 FLOURENES		6.4	11.3	20.1	35.3	61.3	85.7	100.0	89.0
7 PHENANTHRENES		5.3	10.2	21.4	35.8	54.9	69.0	75.2	74.6
8									

0 TYPE OF RING	10 C31	11 C32	12 C33	13 C34	14 C35	15 C36	16 C37	17 C38
1 ALKYLBENZENES	89.2	85.2	77.3	68.9	61.3	52.7	44.4	37.2
2 BENZOCYCLOPARAFFINS	98.4	94.9	88.9	79.9	71.7	61.6	57.1	44.1
3 BENZODICYCLOPARAFFINS	83.7	84.2	80.0	73.5	64.9	56.7	48.7	40.7
4 NAPTHALENES	88.8	85.1	80.6	70.8	63.2	53.5	46.7	37.8
5 NAPHTHOCYCLOPARAFFINS	91.4	88.0	83.0	73.6	64.2	54.2	46.5	38.3
6 FLOURENES	84.0	84.6	80.5	69.6	61.3	51.0	43.1	35.0
7 PHENANTHRENES	76.1	71.4	66.7	57.9	51.8	45.0	38.4	31.8
8								

Table 12. (Cont.)

880331

0 TYPE OF RING	18 C39	19 C40	20 C41	21 C42	22 C43	23 C44	24 C45	25 C46
1 ALKYLBENZENES	30.4	21.7	15.6	10.8	6.7	3.7	2.0	0.8
2 BENZOCYCLOPARAFFINS	36.3	26.8	19.0	13.6	8.5	5.3	2.9	1.3
3 BENZODICYCLOPARAFFINS	33.4	26.3	18.2	13.1	8.2	5.0	2.7	1.3
4 NAPTHALENES	31.0	24.1	16.6	11.8	7.5	4.6	2.4	1.0
5 NAPHTHOCYCLOPARAFFINS	31.1	25.1	16.4	11.9	7.4	4.4	2.4	1.0
6 FLOURENES	29.5	23.5	15.7	11.1	7.2	4.0	2.1	0.9
7 PHENANTHRENES	26.8	22.2	14.5	10.4	7.0	3.8	2.2	0.9
8								

0 TYPE OF RING	26 C47	27
1 ALKYLBENZENES	0.2	
2 BENZOCYCLOPARAFFINS	0.3	
3 BENZODICYCLOPARAFFINS	0.4	
4 NAPTHALENES	0.2	
5 NAPHTHOCYCLOPARAFFINS	0.2	
6 FLOURENES	0.3	
7 PHENANTHRENES	0.2	
8		

Table 12. (Cont.)

880608

0 Type of Ring	1 C22	2 C23	3 C24	4 C25	5 C26	6 C27	7 C28	8 C29	9 C30
1 Alkylbenzenes	15.8	22.9	35.2	50.2	64.9	75.5	79.0	78.6	88.6
2 Benzocycloparaffins	13.9	22.2	31.4	43.8	57.3	68.8	75.4	82.1	86.9
3 BENZODICYCLOPARAFFINS		8.2	14.6	24.1	37.5	50.0	61.3	69.6	73.2
4 NAPTHALENES		5.3	13.8	29.3	47.7	66.5	75.3	82.1	78.6
5 NAPTHOCYCLOPARAFFINS		9.4	18.4	31.8	49.4	66.4	79.2	87.5	82.7
6 FLOURENES		6.1	14.0	27.9	47.1	74.4	92.4	100.0	82.4
7 PHENANTHRENES		6.2	15.1	30.0	45.9	62.7	73.2	75.1	70.3
8									
9									

0 Type of Ring	10 C31	11 C32	12 C33	13 C34	14 C35	15 C36	16 C37	17 C38
1 Alkylbenzenes	79.3	70.8	62.7	53.6	46.7	40.1	33.9	28.1
2 Benzocycloparaffins	85.4	77.5	68.0	59.3	52.3	45.2	42.7	32.6
3 BENZODICYCLOPARAFFINS	70.0	65.1	59.2	51.9	45.9	40.0	34.6	28.5
4 NAPTHALENES	72.3	65.7	58.7	49.5	44.3	37.7	33.4	26.0
5 NAPTHOCYCLOPARAFFINS	76.9	71.0	64.6	55.5	47.8	41.1	34.3	28.2
6 FLOURENES	74.6	73.6	68.9	56.8	48.9	39.8	33.6	27.3
7 PHENANTHRENES	69.6	62.3	56.8	47.8	41.9	36.4	30.4	25.3
8								
9								

Table 12. (Cont.)

880608

0 Type of Ring	18 C39	19 C40	20 C41	21 C42	22 C43	23 C44	24 C45	25 C46
1 Alkylbenzenes	23.2	16.6	11.7	8.0	4.6	2.5	1.1	0.3
2 Benzocycloparaffins	27.0	20.1	14.2	9.6	5.8	3.3	1.5	0.6
3 BENZODICYCLOPARAFFINS	23.2	17.6	12.4	8.5	4.9	2.6	1.3	0.4
4 NAPTHALENES	21.5	15.9	11.0	7.4	4.2	2.3	0.9	0.1
5 NAPTHOCYCLOPARAFFINS	22.5	17.3	11.4	7.8	4.5	2.3	0.9	0.2
6 FLOURENES	22.5	17.5	11.6	8.1	4.8	2.5	1.1	0.2
7 PHENANTHRENES	20.9	16.8	11.3	7.6	4.9	2.4	1.1	0.2
8								
9								

0 Type of Ring	26	27	28	29	30	31	32	33	34	35
1 Alkylbenzenes										
2 Benzocycloparaffins										
3 BENZODICYCLOPARAFFINS										
4 NAPTHALENES										
5 NAPTHOCYCLOPARAFFINS										
6 FLOURENES										
7 PHENANTHRENES										
8										
9										

Table 12. (Cont.)

880701

0 TYPE OF RING	1 C22	2 C23	3 C24	4 C25	5 C26	6 C27	7 C28	8 C29	9 C30
1 ALKYLBENZENES	14.2	24.4	35.3	52.5	67.8	78.2	81.3	82.6	92.5
2 BENZOCYCLOPARAFFINS	18.5	28.1	36.4	49.7	60.8	70.7	77.3	83.9	89.6
3 BENZODICYCLOPARAFFINS		14.0	20.8	29.5	41.1	52.4	61.6	69.7	73.7
4 NAPTHALENES		9.0	16.9	32.3	52.6	69.5	77.1	83.0	82.4
5 NAPHTHOCYCLOPARAFFINS		11.2	21.0	34.8	54.0	68.8	82.2	91.0	89.0
6 FLOURENES		10.1	18.2	29.6	47.2	71.8	90.6	100.0	86.1
7 PHENANTHRENES		7.1	14.8	28.1	44.1	62.2	74.0	77.4	74.6
8									

0 TYPE OF RING	10 C31	11 C32	12 C33	13 C34	14 C35	15 C36	16 C37	17 C38
1 ALKYLBENZENES	85.1	75.8	65.4	56.9	48.3	40.6	33.4	27.6
2 BENZOCYCLOPARAFFINS	87.7	78.7	69.6	60.6	52.8	44.3	40.5	30.2
3 BENZODICYCLOPARAFFINS	71.4	67.3	60.6	53.1	46.3	38.9	32.8	26.6
4 NAPTHALENES	77.4	70.1	62.3	53.9	46.4	37.9	32.0	25.0
5 NAPHTHOCYCLOPARAFFINS	84.0	77.1	69.4	58.9	50.1	41.6	33.6	27.4
6 FLOURENES	79.0	77.2	71.6	59.5	50.6	40.2	33.2	26.1
7 PHENANTHRENES	74.9	68.1	60.4	51.2	43.9	37.3	30.5	25.1
8								

Table 12. (Cont.)

880701

0 TYPE OF RING	18 C39	19 C40	20 C41	21 C42	22 C43	23 C44	24 C45	25 C46
1 ALKYLBENZENES	21.8	15.1	10.8	7.4	4.3	2.4	1.2	0.4
2 BENZOCYCLOPARAFFINS	24.6	17.5	12.3	8.6	5.0	2.9	1.4	0.6
3 BENZODICYCLOPARAFFINS	21.0	15.0	10.7	7.4	4.1	2.3	1.1	0.3
4 NAPTHALENES	20.0	14.0	9.7	6.3	3.6	2.0	0.8	0.2
5 NAPHTHOCYCLOPARAFFINS	21.0	15.9	10.3	7.1	4.0	2.2	0.9	0.2
6 FLOURENES	21.1	15.8	10.4	7.3	4.4	2.2	1.1	0.3
7 PHENANTHRENES	20.1	15.7	10.4	7.2	4.7	2.4	1.2	0.4
8								

0 TYPE OF RING	26
1 ALKYLBENZENES	
2 BENZOCYCLOPARAFFINS	
3 BENZODICYCLOPARAFFINS	
4 NAPTHALENES	
5 NAPHTHOCYCLOPARAFFINS	
6 FLOURENES	
7 PHENANTHRENES	
8	

Table 12.

OIL SANDS AND HYDROCARBON RECOVERY DEPARTMENT

VALUES AND PRINCIPLES

AS A LEADER IN OIL SANDS AND HYDROCARBON RESEARCH WE ARE PROUD OF OUR REPUTATION FOR:

- Developing and improving technology through applied research.
- Facilitating the transfer of technology and providing information, expert advice, and related scientific infrastructure to the private and public sectors.

WE BELIEVE OUR STRENGTHS ARISE FROM A COMMITMENT TO:

- **Excellence,** in maintaining a position at the forefront of science and technology; in serving the needs of our clients; in forming productive relationships with industry, universities and sister organizations.
- **People,** who are the foundation of our success; and to an environment of mutual trust and respect which recognizes the importance of health, safety, personal development and fairness as equally important as our research and development objectives.
- **Teamwork,** by recognizing the participation and contribution of all staff members in achieving our objectives; and by encouraging a supportive environment that fosters open communications, cooperation and creativity.

OUR SUCCESS IS BASED ON THE JOINT COMMITMENT OF EVERYONE TO THE FOLLOWING VALUES:

Excellence: We recognize the significance of excellence for continued success, and the need for initiative, effort, and creativity to broaden our reputation as a leader in our field.

Mutual Respect: We only ask of others what we would be willing to do ourselves. We are considerate of people's time, opinions, feelings, personal beliefs and needs.

Open Communication: Within the constraints of the Alberta Research Council/client contractual agreements, information is freely shared among staff members. Informal avenues of communication are encouraged. Stakeholders are involved wherever possible before decisions are made.

Recognition: Everyone receives timely and regular feedback, both positive and negative, for his/her efforts and achievements.

Honesty: Consistent with the highly professional nature of our work we are fastidious in our regard for honesty in reporting scientific results and in all our dealings with each other and with others outside the department.

Teamwork: We recognize the value of individual effort but also recognize the importance of cooperation, collaboration and the participation of everyone in achieving common goals. Accordingly, we contribute willingly to team efforts and value the contributions of all team members.

Health: We believe in a work environment that promotes the health and well-being of each individual.

Safety: Safety is everyone's responsibility. We believe in safety and are conscientious in our efforts to incorporate safe working practices in all our activities, to comply with all safety regulations and procedures and the implementation of necessary safety practices.

Staff Development: People are the foundation of our success. We recognize the need to remain at the forefront of knowledge and endeavour to provide all staff with the opportunities to continue their personal and career growth while fulfilling Oil Sands and Hydrocarbon Recovery Department's needs.

Trust: We recognize the importance of trust as the building block for all our relationships and reflect this trust in our organizational structures, administrative practices, delegation of responsibility and authority, dealings with each other, and accountability for assigned responsibilities.

Fairness: We believe in the fair and equitable treatment of all staff and the conduct of our business consistent with Alberta Research Council's code of ethics.

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